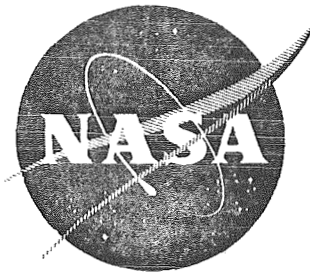


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## ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

### QUARTERLY PROGRESS REPORT NO. 18 For Quarter Ending October 15, 1969

prepared by  
R. W. Harrison

prepared for  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center  
Contract NAS 3-6474  
R. L. Davies and P. L. Stone, Project Managers  
Materials Section

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QUARTERLY PROGRESS REPORT 18

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

prepared by  
R. W. Harrison

approved by  
E. E. Hoffman

NUCLEAR SYSTEMS PROGRAMS  
SPACE SYSTEMS  
GENERAL ELECTRIC COMPANY  
Cincinnati, Ohio 45215

prepared for  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Period July 15 to October 15, 1969

October 23, 1969

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NASA Lewis Research Center  
Cleveland, Ohio  
R. L. Davies and P. L. Stone, Project Managers  
Materials Section

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## FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies and P. L. Stone of NASA-Lewis Research Center are the NASA Technical Managers.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop Operation - J. Holowach, A. Losekamp, T. Irwin

Partial Pressure Gas Analysis - Dr. T. Lyon

1900°F Lithium Loop - J. Smith

Advanced Tantalum Alloy Capsule Tests - G. Brandenburg



## ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

### I. INTRODUCTION

This report covers the period from July 15, 1969 to October 15, 1969. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which has been tested under Contract NAS 3-2547.<sup>(1)</sup> Lithium is being heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop is being accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

- a. Boiling temperature, 2050°F.
- b. Superheat temperature, 2150°F.
- c. Condensing temperature, 1400°F.
- d. Subcooling temperature, 1000°F.
- e. Mass flow rate, 40 lb/hr
- f. Boiler exit vapor velocity, 50 ft/sec
- g. Average heat flux in plug (0-18 inches), 240,000 Btu/hr ft<sup>2</sup>
- h. Average heat flux in boiler (0-250 inches), 23,000 Btu/hr ft<sup>2</sup>

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium.

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<sup>(1)</sup> Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

Also included in the program is the fabrication, 7500-hour operation and evaluation of a 1900°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of T-111 clad fuel specimens, ASTAR 811 type alloys, T-111, T-222, and the tungsten alloy, W-25Re-3OMo at conditions simulating a lunar Brayton reactor system.

## II. SUMMARY

On October 3, 1969 the T-111 Rankine System Corrosion Test Loop had successfully completed 6000 hours of operation. The loop was put on automatic control and constant surveillance was terminated.

Posttest evaluation of the lithium thermal convection capsules revealed corrosion in ASTAR 811C specimens which had been welded and not postweld annealed.

Fabrication of the 1900°F Lithium Loop is nearing completion. All subassemblies have been joined.



III. PROGRAM STATUSA. T-111 RANKINE SYSTEM CORROSION TEST LOOP1. Loop Operating Temperatures

On August 22, 1969 the T-111 Corrosion Test Loop successfully completed the first half of the planned 10,000 hours of operation. The loop temperatures recorded at the 5000 hour test time are shown in Figure 1, and the temperatures of major interest are shown on the loop schematic in Figure 2. The performance of the loop has been excellent in that control adjustments have been limited to minor changes in the power input to the lithium heater to compensate for small line voltage changes not compensated for by the voltage stabilizer. A comparison of the data obtained at 1000 hours, 2000 hours, and 5000 hours shown in Table I, further exemplifies the stability of the loop's performance.

2. Turbine Simulator Performance

The calculated vapor velocities of the turbine simulator nozzles at 5000 hours are presented in Table II for a mass velocity of 36.7 lb/hr of potassium. The vapor velocity in the superheated first stage was 1080 ft/sec. The vapor velocity in the 88-percent-quality region ranged from a high of 1280 ft/sec in the second-stage nozzle to a low of 1150 ft/sec in the tenth stage nozzle. All vapor velocities were higher than the 1000 ft/sec design velocity.

The higher than design velocity is attributed to the lower than predicated vapor pressure at the inlet to the turbine simulator due to a higher than predicated pressure drop in the boiler. The higher than predicated pressure loss in the boiler is due to the high heat transfer rate in the 18-inch-long plug and the resulting high vapor quality in the entrance section of the boiler. For a given mass flow rate, the pressure drop in the tube is inversely proportional to the vapor density.

3. Test Chamber Environment-Partial Pressure Analysis

The chamber pressure and partial pressures of the various gaseous species in the test chamber during the period from 4000 to 6000 hours of loop operation are shown in Figure 3. Similar data for the period from 0 to 4000 hours of loop operation were given in the preceding

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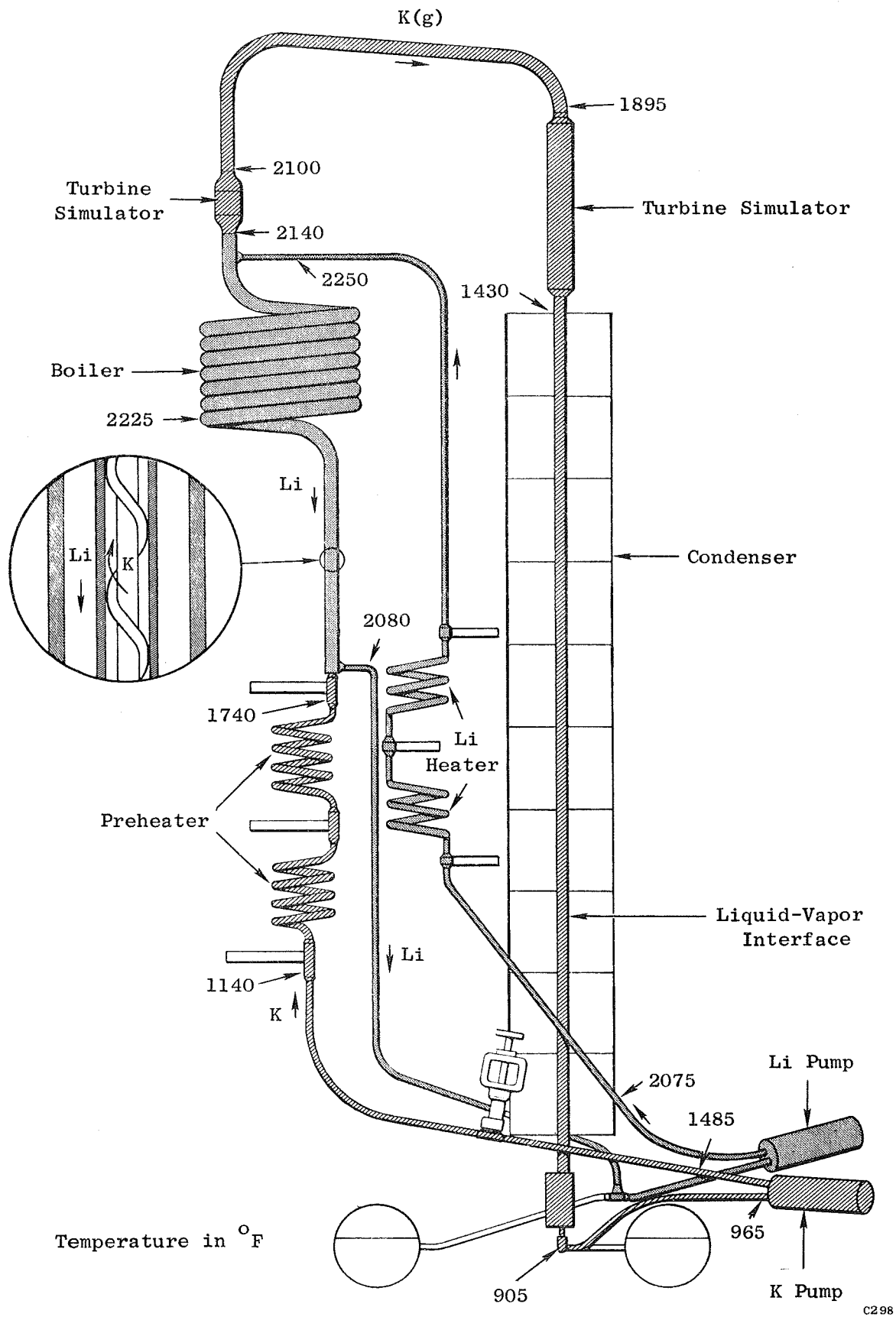


Figure 2. T-111 Corrosion Test Loop Operating Temperatures - 5000 Hours.

TABLE I

## T-111 RANKINE SYSTEM CORROSION TEST LOOP PERFORMANCE

<u>Date</u>	3-8-69	4-19-69	8-22-69
<u>Test Hours</u>	1000	2000	5000
Lithium Flow Rate	205 lbs/hr	207 lbs/hr	229 lbs/hr
Lithium Temperature, In	2253°F	2253°F	2239°F
Lithium Temperature, Out	2078°F	2078°F	2078°F
Lithium $\Delta T$	175°F	175°F	161°F
Potassium Flow Rate	36 lbs/hr	37 lbs/hr	38 lbs/hr
Plug Boiling Temperature	2048°F	2048°F	2052°F
Boiler Exit Vapor Temp.	2147°F	2138°F	2137°F
Boiler Exit Saturation Temp.	2012°F	2011°F	2012°F
Potassium Vapor Superheat	135°F	127°F	125°F
Condensing Temperature	1416°F	1416°F	1411°F
<u>Potassium Heat Input</u>			
1. Preheat	2280 Btu/hr	2340 Btu/hr	2410 Btu/hr
2. Heat of Vaporization	26,300 Btu/hr	26,950 Btu/hr	27,600 Btu/hr
3. Superheat	1040 Btu/hr	947 Btu/hr	960 Btu/hr
TOTAL	29,620 Btu/hr	30,237 Btu/hr	30,970 Btu/hr
Total Power to Lithium Heater	13.2 kw	13.7 kw	13.8 kw
Total Power to Potassium	8.7 kw	8.9 kw	9.0 kw
Net Heat Loss	4.5 kw	4.8 kw	4.8 kw



TABLE II

T-111 RANKINE SYSTEM CORROSION TEST LOOP  
TURBINE SIMULATOR PERFORMANCE AT 5000 HOURS

Nozzle Number	Material	Nozzle Diameter <sup>(a)</sup> inch	Inlet Temperature °F	Exit Pressure, psia	Vapor Velocity ft/sec
1	Mo-TZC	0.0892	2137	112.4	1080
2	Mo-TZC	0.0881	1899	92.5	1280
3	Mo-TZC	0.0964	1839	75.0	1280
4	Mo-TZC	0.1083	1777	61.0	1240
5	Mo-TZC	0.1181	1720	49.5	1260
6	Cb-132M	0.1292	1664	40.0	1280
7	Mo-TZC	0.1457	1611	32.5	1215
8	Mo-TZC	0.1598	1562	26.3	1230
9	Cb-132M	0.1784	1514	21.5	1190
10	Mo-TZC	0.1986	1471	17.6	1150

(a) Measured at room temperature

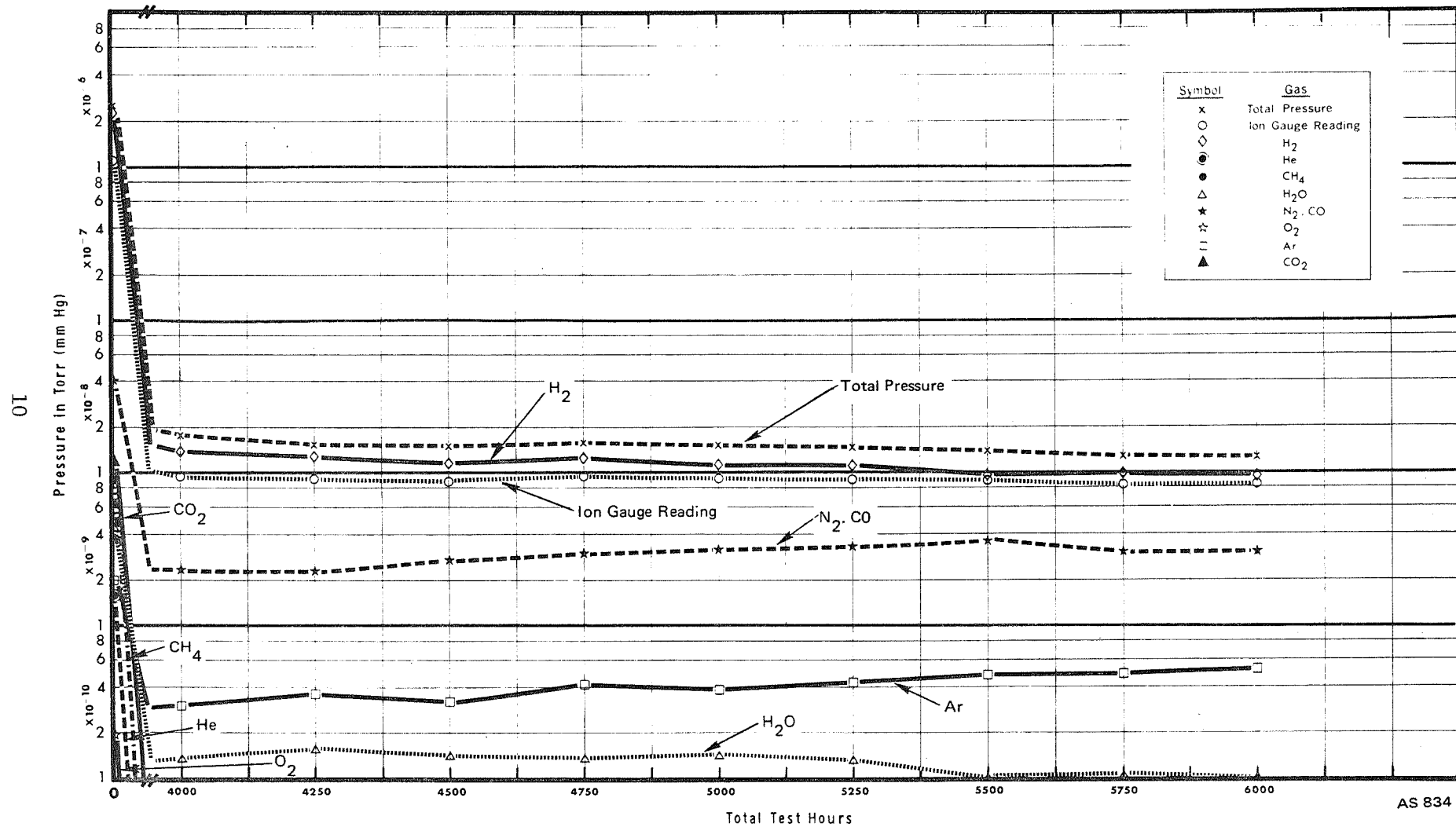


Figure 3. Test Chamber Environment During Testing of the T-111 Rankine System Corrosion Test Loop.

quarterly reports.<sup>(1,2)</sup> In these Figures, data points are plotted at 250 hour intervals for the sake of clarity, even though residual gas analyses are obtained every eight hours during loop operation.

As shown in Figure 3, the total pressure has gradually decreased over this time interval as a result of the diminishing hydrogen partial pressure. Hydrogen has been and continues to be the predominant gas in the system.

#### 4. Loop Operation

As a result of the stable and trouble-free operation of the loop during the first 5000 hours of operation the need for constant surveillance was re-evaluated.

As described previously (A1), during constant surveillance operation adjustments to the loop controls were limited to manual adjustments to the lithium heater power control, a General Electric Type 524 Current-Modulation Controller. This unit is capable of automatically controlling the power to maintain a specific temperature when used in conjunction with a General Electric Type HF temperature recorder. On September 29, 1969 the lithium heater power was placed on automatic control.

Appropriate adjustments to the control band width resulted in control of the lithium heater exit temperature within a 20°F band at 2250°F with no difficulties. On October 3, 1969 the loop had completed 6000 hours of operation, and surveillance was reduced to one shift for data taking purposes only since no requirement for adjustments was anticipated. On October 23, 1969 the loop completed 6500 hours of operation and 500 hours on automatic control.

Automatic and unattended operation of a corrosion loop of this complexity can be considered a major accomplishment and is only possible due to the stable and trouble-free operation of the T-111 Rankine System Corrosion Test Loop demonstrated to date.

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 16 for Period Ending April 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72560.

(2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 17 for Period Ending July 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72592.

## B. 1900°F LITHIUM LOOP

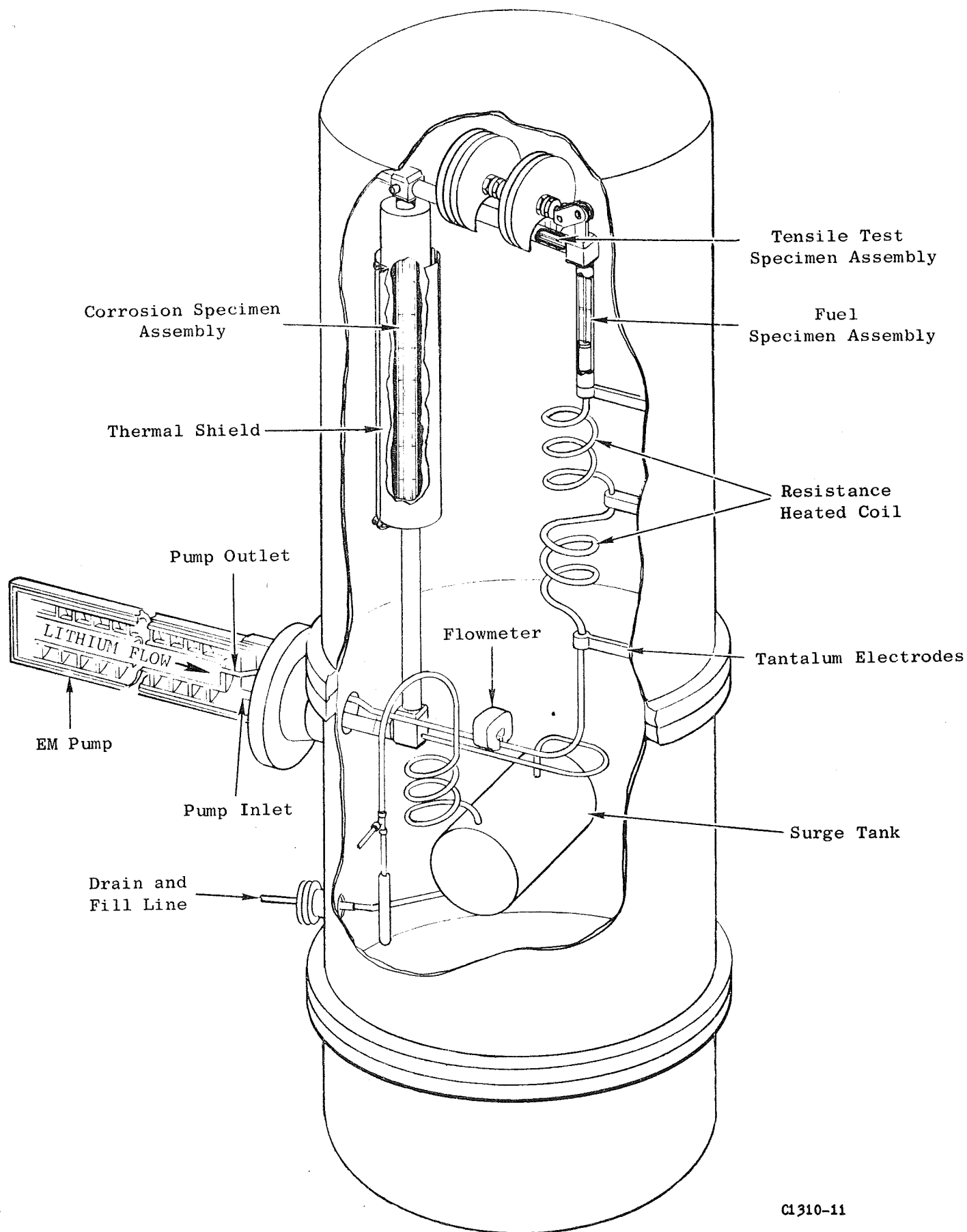
Fabrication of all subcomponents for the 1900°F Lithium Loop has been completed. The heater electrode subassembly; corrosion, tensile and clad fuel element subassemblies were welded together thus completing a major portion of the loop assembly. The relative position and orientation of these subassemblies in the loop is illustrated in the isometric drawing, Figure 4. All welds are made according to GE-NSP Specification 03-0025-00-A. Final welding of the EM pump and surge tank into the loop will be performed after completion of postweld annealing of all welds performed to date.

Prior to assembly of the subcomponents, all specimens were cleaned and weighed accurately to obtain data suitable for weight change measurements at the completion of the test. The T-111 clad fuel element specimens which were received from NASA on August 12, 1969 were radiographed to determine the integrity of the encapsulated fuel and liners. These radiographs revealed cracking of some of the tungsten dome-shaped end spacers; however, it was agreed by NASA and GE that the cracks will not adversely affect the initial 2500 hours of the experiment since the clad contains no defects. Plans are being made by NASA to provide design or material revisions for the spacers to be used during the next 5000 hours of the experiment. The replacement fuel element specimen will contain a purposely defective clad; hence, the condition of the clad interior is much more critical. It is felt the damage currently observed is due to the inherently brittle nature of tungsten and not because of mishandling during processing. Other than this minor problem, no unusual problems were encountered during loop fabrication and assembly.

Details of the assembly of the various subcomponents are described in the following paragraphs.

### 1. Fuel Test Specimen Capsule Subassembly

Components of the fuel test subassembly are shown in Figure 5. All hardware, including the fuel element cladding, is T-111 except for the Mo-TZM spacers. These spacers serve a dual purpose in that they also will be evaluated for their corrosion resistance. The end caps and containment tube are designed so that the caps fit inside the tube about



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Figure 4. Isometric Drawing of 1900°F Lithium Loop.

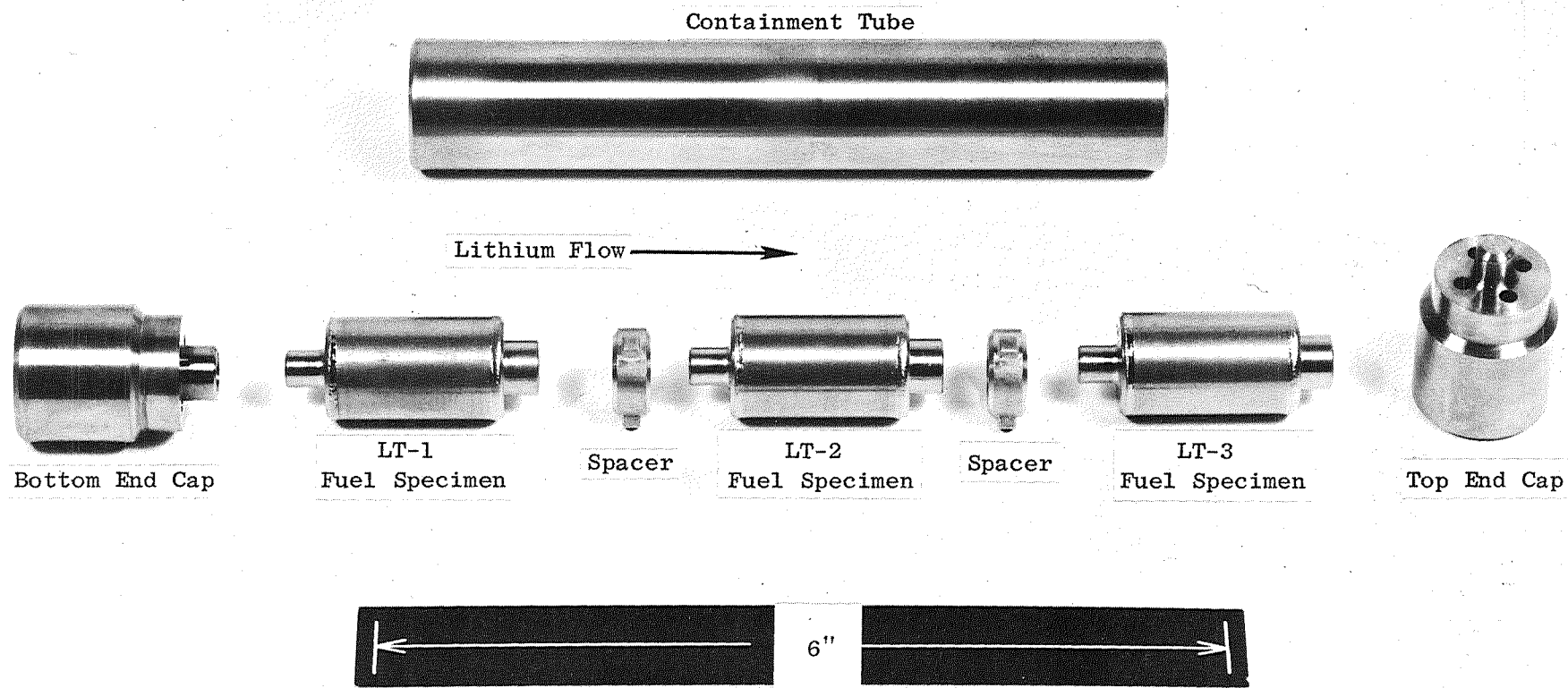


Figure 5. Components of Clad Fuel Element Specimen Capsule Subassembly - 1900°F Lithium Loop. Containment Tube, Caps and Specimen Clad: T-111. Spacers: Mo-TZM.

one-quarter inch and butt against a recess (not visible in Figure 5) on the ID of the tube. This was done to prevent the end caps from applying a compressive load on the fuel specimens during welding shrinkage; instead the loads are supported by the seat in containment tube. The fuel specimens are interlocking in that the male end of one specimens fits into the female end of the other specimen. The interlocking feature of the fuel specimens and Mo-TZM spacers provides the necessary uniform gap between the fuel specimen and the ID of the containment tube to produce the required 5 ft/sec lithium flow in this test section. It also facilitates easy removal of selected specimens at the end of the first 2500 hours operation for replacement with a defective clad specimen. Since the fuel specimens are solid, all lithium flow is along this narrow annulus between the specimen and the containment tube.

This subassembly is situated vertically in the loop, the bottom cap being welded directly to the heater assembly and the top cap welded to the tensile section described in the following paragraph.

## 2. Tensile Test Specimen Subassembly

Components of the tensile specimen subassembly are shown in Figure 6. All material, except the specimens, is T-111 alloy. This subassembly consists of three individual specimen holders each of which contains two (except for the W-Re-Mo alloy) 0.040 inch thick coupons held within close-fitting slots on the inside and two specimens strapped to the outside flats shown in Figure 7. The W-25Re-30Mo\* coupons are only 0.020 inch thick; therefore, each slot contains two coupons instead of one for a total of four specimens in that particular specimen holder. The gap between the "inside" specimens is controlled to obtain the desired lithium velocity of 10 ft/sec over one surface of each coupon. The coupons strapped to the outside of each specimen holder will be used as control specimens since they see the same thermal history as the inside coupons but are not exposed to lithium. The holes in the "inside" coupons, as shown in Figure 7, are for locating pins to prevent movement during the high-velocity lithium flow.

\* Atomic percent

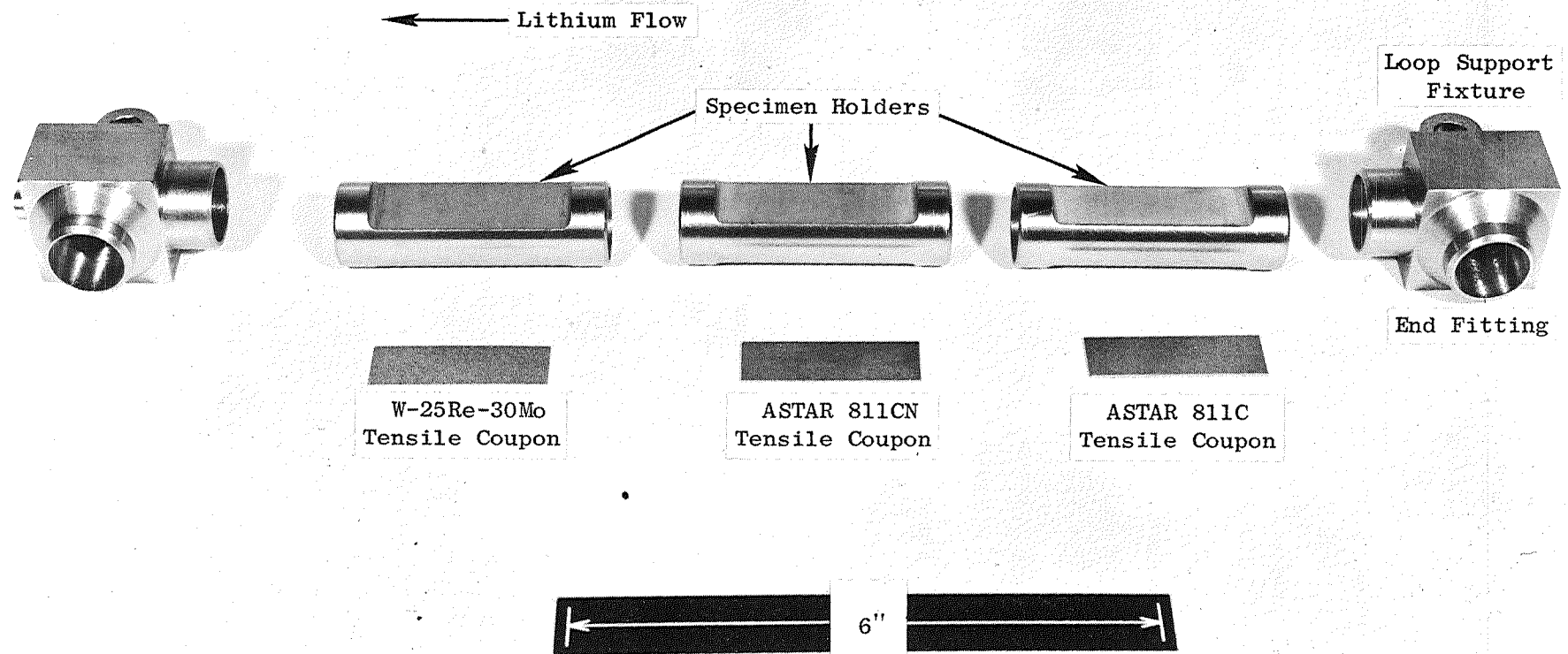


Figure 6. Components of Tensile Test Specimen Subassembly - 1900°F Lithium Loop.  
Specimen Holder and Fittings: T-111.



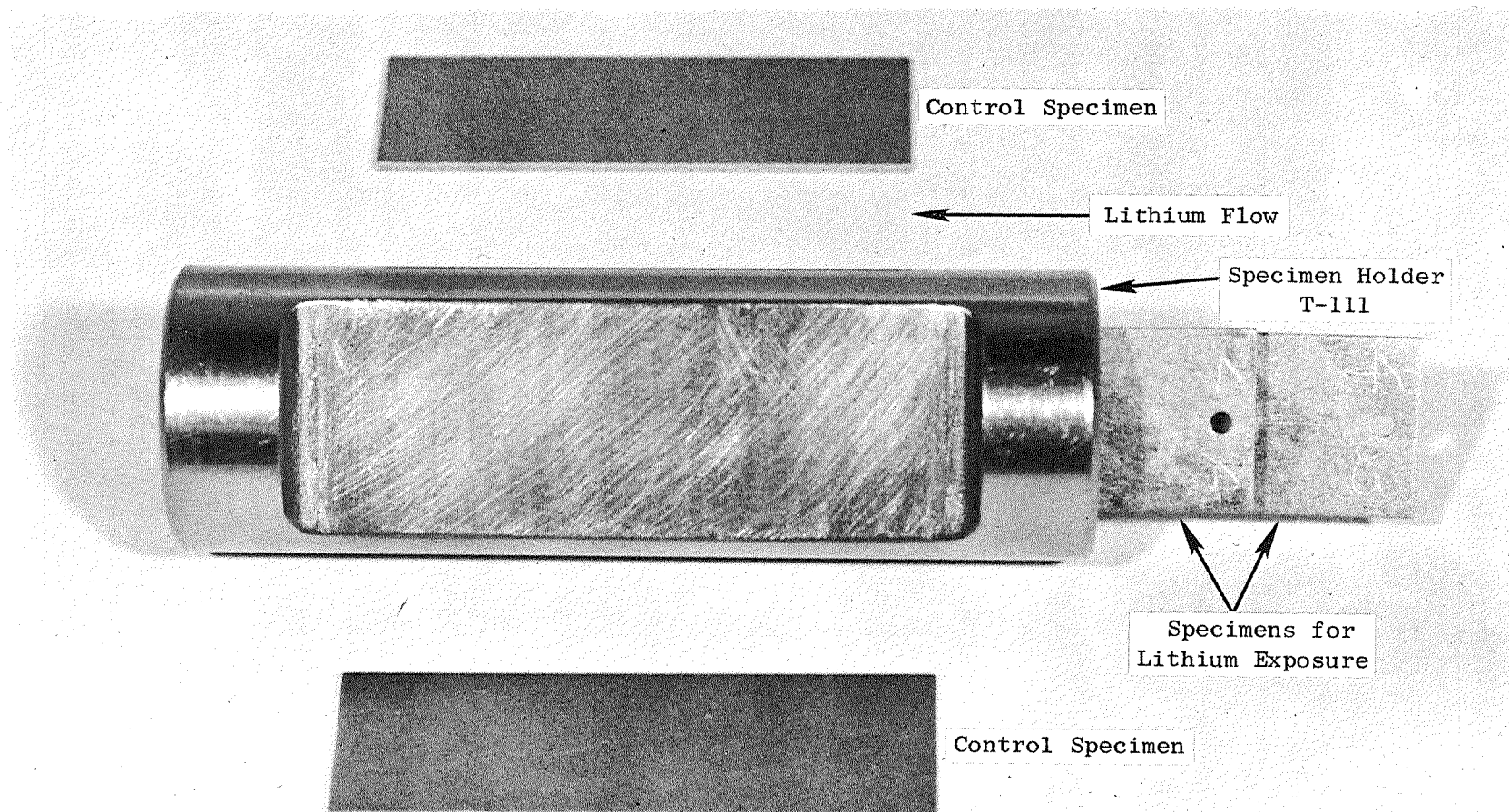


Figure 7. Assembly of Tensile Test Specimen Holder - 1900°F Lithium Loop.

On August 8, 1969, a heat treatment of one-half hour at 3600°F was selected by NASA for the ASTAR 811C specimens. The specimens were subsequently annealed in an ion-pumped vacuum facility capable of a cold wall vacuum of  $1 \times 10^{-9}$  torr. Chemical analysis of an ASTAR 811C specimen before and after the anneal indicated no major change in interstitial concentrations as shown in Table III.

The sequence of fabrication for the tensile specimen subassembly was as follows: (1) insert inside specimen coupons and locating pins in each of the three specimen holders, (2) weld the three holders together, and (3) weld the end fitting and blow-down tube to the specimen holders. The completed subassembly is shown in Figure 8. The "outside" specimen coupons were attached after assembly welding of the various subcomponents and will be included in the postweld anneal.

The fixtures at the top of the end fitting shown in Figure 6 are used to hang the loop in the support structure during test operation. The blow-down tube is used to aid removal of lithium at the completion of the test. All specimen coupons will be cut into standard tensile test specimen at the completion of the 7500 hours of testing for post-test evaluation.

### 3. Corrosion Test Specimen Subassembly

All components and specimens for the corrosion test specimen subassembly are shown in Figure 9. The parts shown were fabricated from T-111 with the exception of the ASTAR 811C and ASTAR 811CN specimens. Because of raw material restrictions for the ASTAR 811CN, it was necessary to make eight disc-shaped pieces and stack these to be equivalent in length to one of the ASTAR 811C and T-111 specimens. A 0.020-inch T-111 wire which was wound around the center rod (two-inch pitch) to improve flow characteristics and to aid in maintaining concentricity is not shown in Figure 9. Because of the number of parts involved and the close fits involved, this was the most difficult subassembly to put together. The sequence of operations was as follows:

- a) Weld top alignment fixture to center rod
- b) Weld bottom alignment fixture to specimen containment tube

TABLE III

CHEMICAL ANALYSES OF VACUUM  
ANNEALED ASTAR 811 C

Element	Concentration, ppm	
	Before Anneal	After Anneal <sup>(a)</sup>
Oxygen <sup>(b)</sup>	10, 13	2, 10
Nitrogen <sup>(b)</sup>	4, 6	2, 3
Carbon <sup>(c)</sup>	237, 249	232, 270
Hydrogen <sup>(b)</sup>	2, 3	1, 1

(a) One-half hour at 3600°F in vacuum  $2 \times 10^{-6}$  torr.

(b) Vacuum Fusion Analysis in duplicate.

(c) Combustion Conductometric Analysis in duplicate.

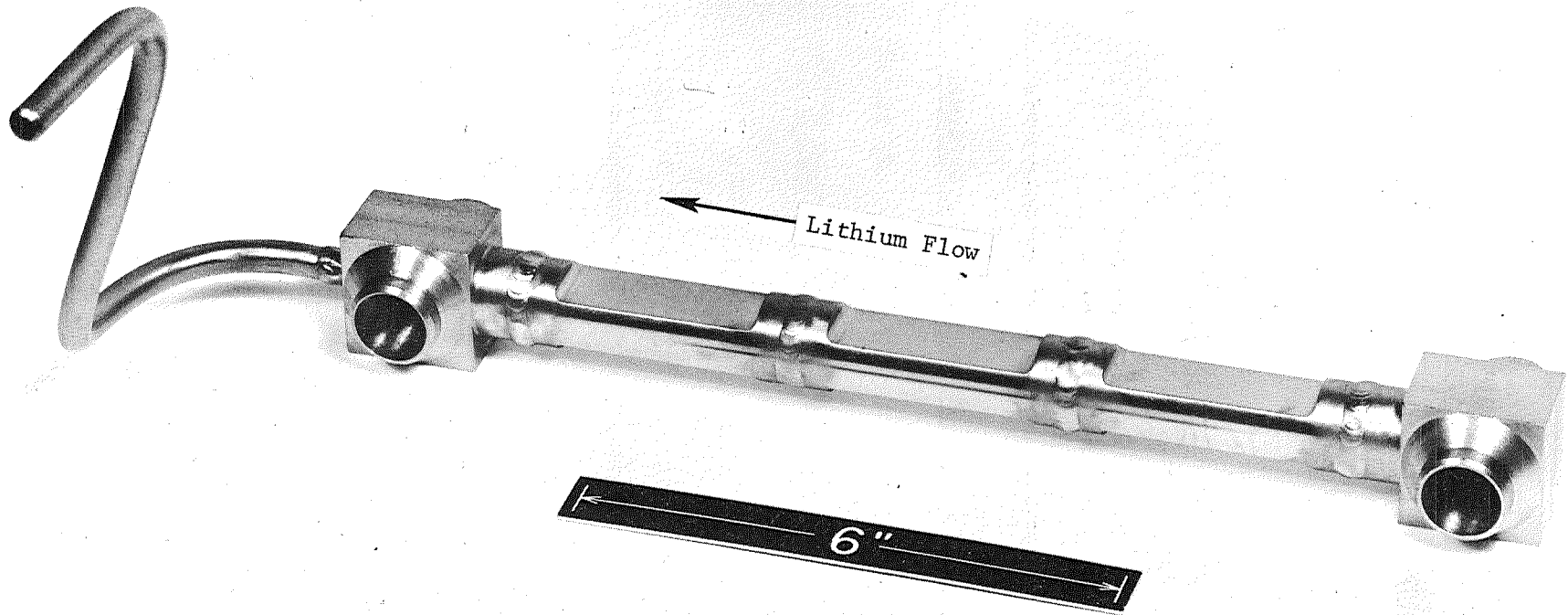


Figure 8. Tensile Test Specimen Subassembly - 1900°F Lithium Loop.

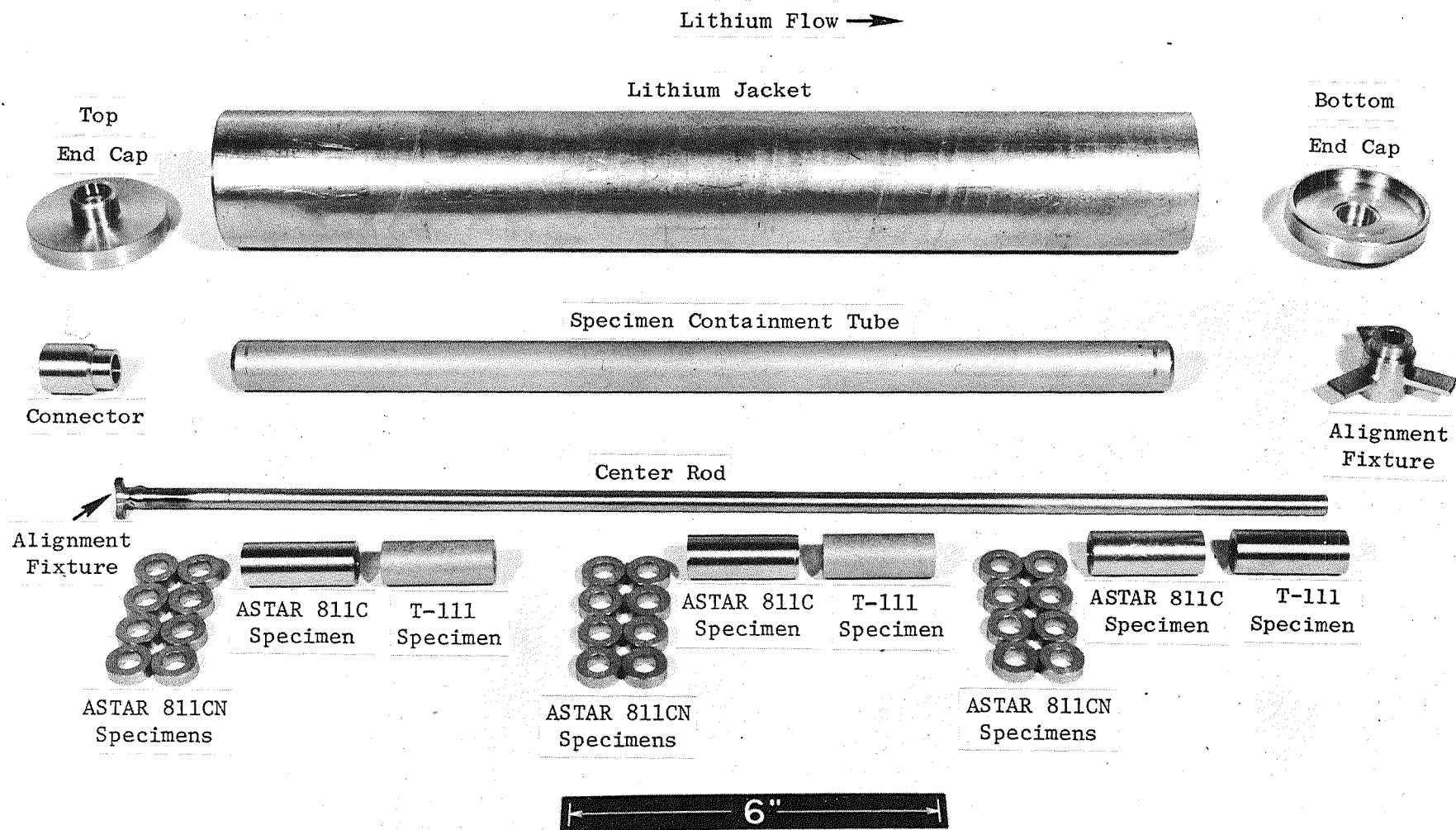


Figure 9. Components of Corrosion Specimen Subassembly - 1900°F Lithium Loop.  
All Components not Designated are T-111.

- c) Spot weld 0.020 inch wire to center rod
- d) Slip connector over center rod
- e) Slip specimens over center rod
- f) Insert specimens and center rod assembly into the specimen containment tube.
- g) Weld specimen containment assembly to connector.
- h) Weld connector, end caps and lithium jacket to complete assembly as shown in the right half of Figure 10.
- i) Finally the extension tube, fitting, and tubes to the EM pump and surge tank were welded as shown in the left half of Figure 10.

This subassembly is designed so that the lithium in the annulus between the ID of the specimens and the center rod flows at 10 ft/sec. Because of the close fit between the OD of the specimen and the ID of the containment tube essentially no flow occurs at this interface. Also, there is a 3/8-inch gap between the exit of the corrosion section (bottom alignment fixture) and the bottom end cap; this allows the cavity between the OD of the specimen containment tube and the ID of the jacket to fill with essentially static lithium. The purpose of the static lithium reservoir is to aid in the heat rejection in this subassembly to provide the necessary 70°F temperature gradient between the lithium entering the corrosion specimen test section and the lithium exiting.

#### 4. Electrical Resistance Heater

The completed heater subassembly consists of two 3/8-inch-diameter tubular T-111 coils welded to tantalum electrodes. In order to increase lithium velocity to the desired 10 ft/sec, a 1/8-inch-diameter T-111 rod is inserted inside the coil windings. As mentioned earlier, the top of this assembly is welded directly to the fuel element test section. The bottom electrode is connected to a 3/8-inch-diameter T-111 tube which in turn will be welded to the exit of the EM pump.

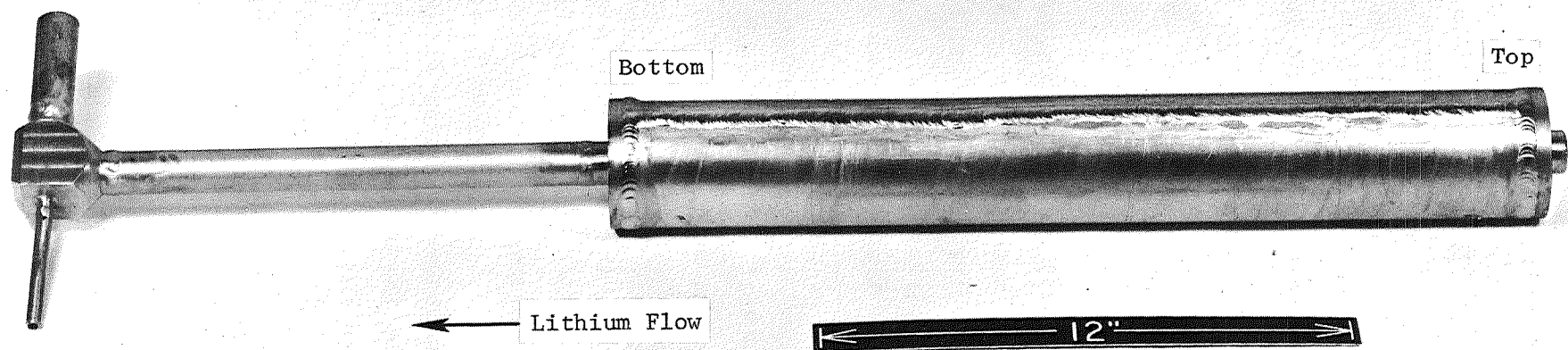


Figure 10. Corrosion Specimen Subassembly, Extension Tube and Fittings to EM Pump - 1900°F Lithium Loop.

C. ADVANCED TANTALUM ALLOY CAPSULE TESTS

1. Posttest Evaluation of Lithium Thermal Convection Capsules

One ASTAR 811CN and two ASTAR 811C lithium thermal convection capsules successfully completed 5000 hours of testing at 2400°F. One ASTAR 811C capsule was retained for possible additional testing. The capsules presently being evaluated are shown in Figure 11 after removal from the test facility.

a. Visual Examination

Examination of the exterior surface of the capsules indicated some discoloration in the heated area of the capsules. Closer examination revealed evidence of vitrification of the BeO insulators and bonding to the capsule surface. A typical capsule after removal of the thermocouple, insulator, and tantalum foil strap in this area, is shown in Figure 12. It is believed that the discoloration is a result of reaction of the BeO insulator with the capsule at temperatures above 2400°F. These insulators have been used extensively at GE-NSP at temperatures below 2300°F without any evidence of reaction. Metallographic techniques will be employed to further examine the capsule wall in the area where reaction was observed although the depth can be considered to be minimal since the discoloration could be removed by light filing.

Prior to opening the capsules radiographs were obtained to delineate the position of the specimens and downcomer tube in each capsule. The radiographs revealed that the downcomer tubes in the ASTAR 811CN capsule and one of the ASTAR 811C capsules had fallen to the bottom of the capsule. From the temperature data obtained on the capsules it is believed that this occurred on the ASTAR 811C capsule after 4500 hours of testing had been accumulated and the ASTAR 811CN capsule in the first 500 hours of testing. The downcomer tube on the ASTAR 811C capsule which was retained for further testing was intact. The thermal data from this capsule was used for the flow calculations previously described. (2)

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(2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 17 for Period Ending July 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72592.



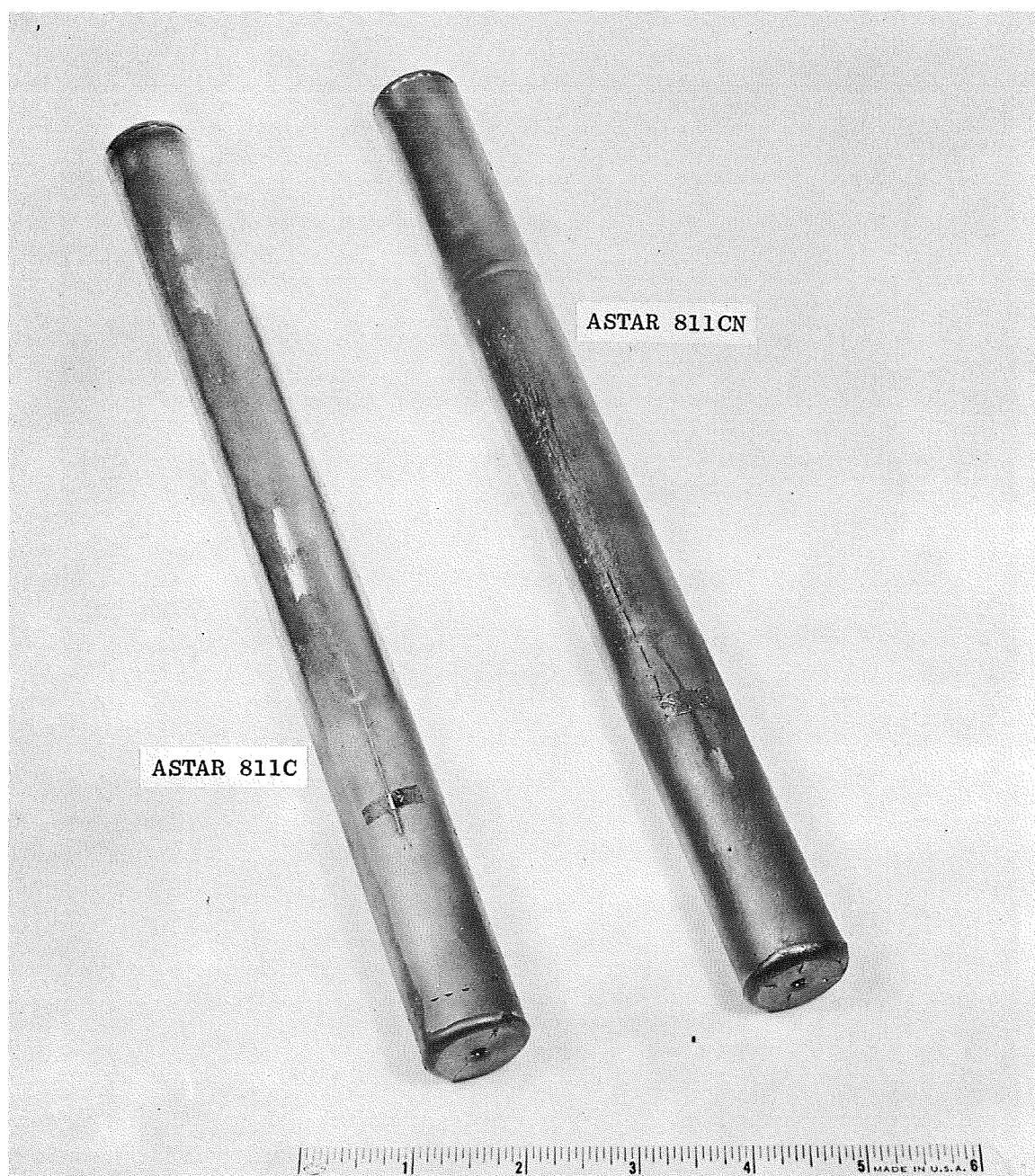


Figure 11. ASTAR 811C and ASTAR 811CN Lithium Thermal Convection Capsules After 5000 Hours of Testing at 2400°F.

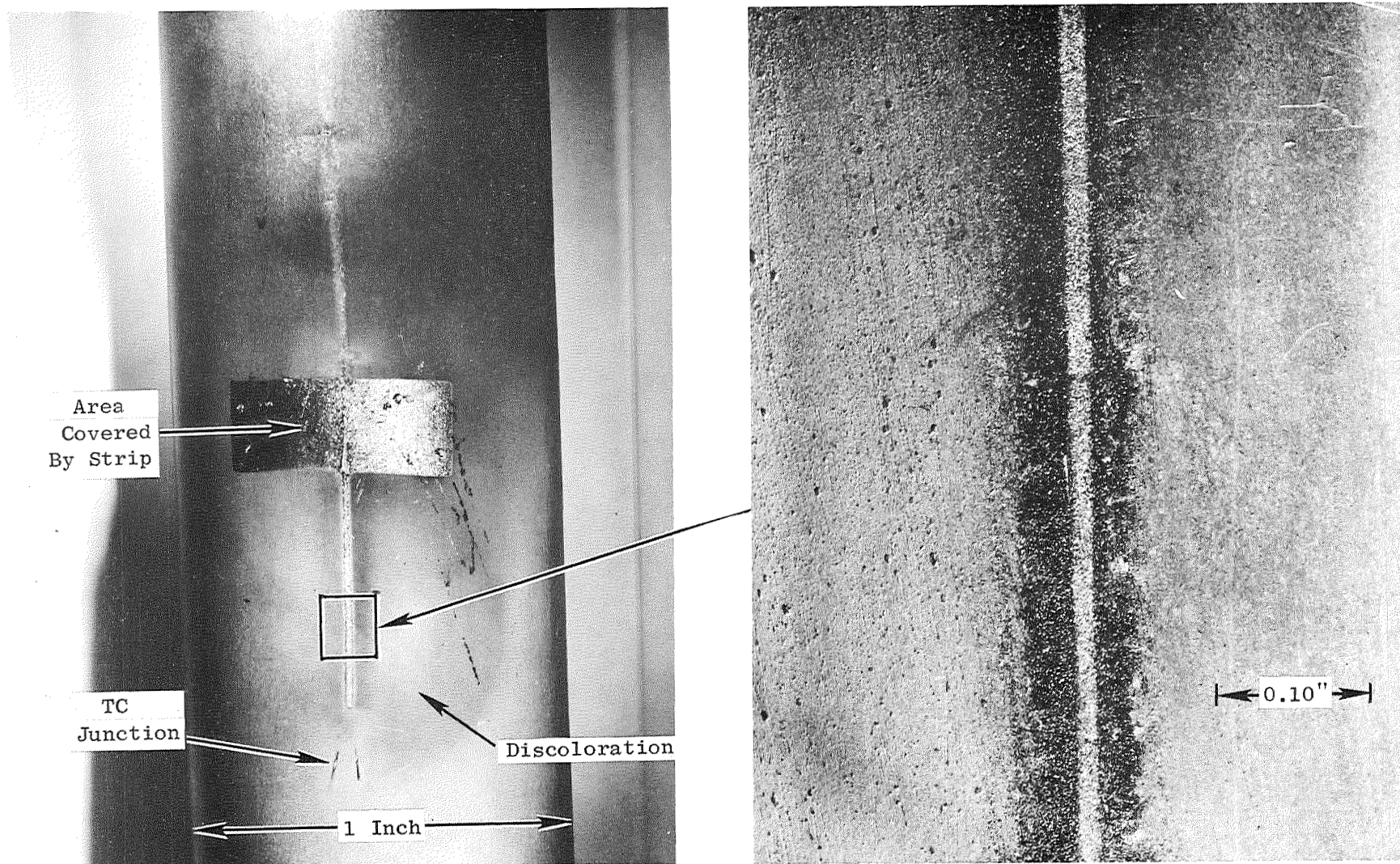


Figure 12. Exterior of ASTAR 811C Capsule Showing the Area Where BeO Insulator Bonded to the Capsule Wall.

The capsules were subsequently opened in a glove box equipped with a recirculating argon purification system capable of maintaining the atmosphere at less than 1 ppm oxygen and less than 1 ppm water vapor. The top of each capsule was removed with a tubing cutter and the capsules were wrapped with heating tapes to melt the lithium. The specimens were removed and the lithium transferred to separate stainless steel sample tubes for retention for chemical analysis. The specimens and capsules were cleaned of residual lithium by reaction with liquid ammonia and rinsed in deionized water.

The disassembled capsules and specimens are shown after cleaning in Figures 13 and 14. The distortion which was observed in the downcomer tubes is believed to be associated with the fact that they dropped to the bottom of the capsules during testing thereby inhibiting flow of the lithium. This type of deformation was not observed in the downcomer tube of the unopened ASTAR 811C capsule radiographs of which showed the downcomer tube to be intact. The design of the downcomer tube support will be modified in future capsule tests of this type.

#### b. Weight Change Measurements

The cleaned specimens were weighted immediately after cleaning and their weights compared with pretest weights. Except for one specimen (R1), the weight change of which is believed in error, all specimens lost weight as shown in Table IV. The weight losses are consistent with metallographic and chemical analysis results which will be described later in this report.

#### c. Bend Tests

The 0.040-inch thick test specimens contained in the ASTAR 811C capsule were bent 90° with the weld face in tension at a strain rate of 0.2 inch per minute using an anvil with a radius of 0.040 inch (1t), to qualitatively document the effects of the lithium exposure on bend ductility. Both pretest and posttest specimens were bent and the results are compared in Table V. All specimens were ductile except specimen 7E, An ASTAR 811C specimen which had been oxygen contaminated and welded prior to exposure to lithium at approximately 2100°F. The slight weld

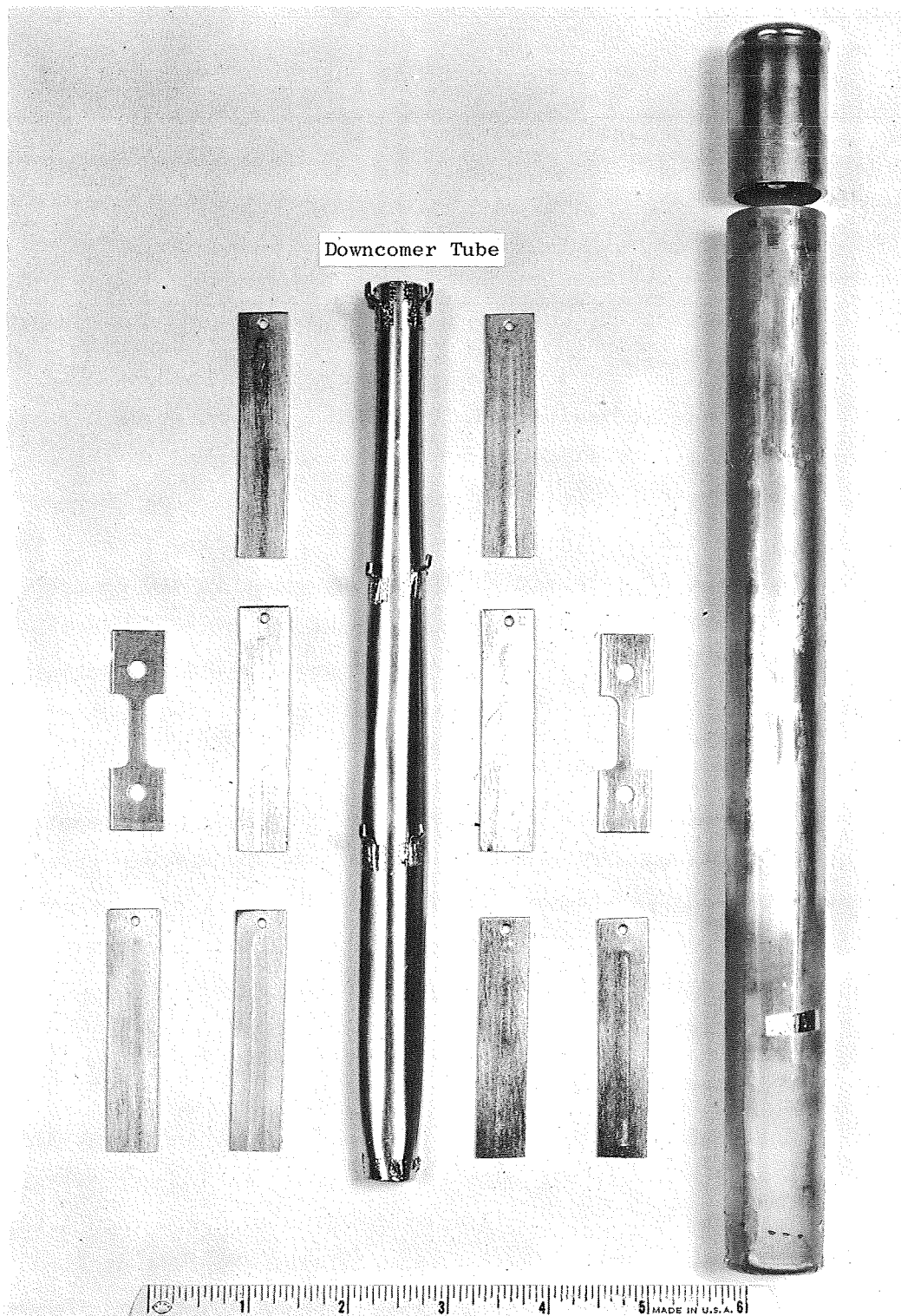


Figure 13. ASTAR 811C Lithium Thermal Convection Capsule Components Following 5000 Hours Exposure to Flowing Lithium at 2400<sup>o</sup>F.



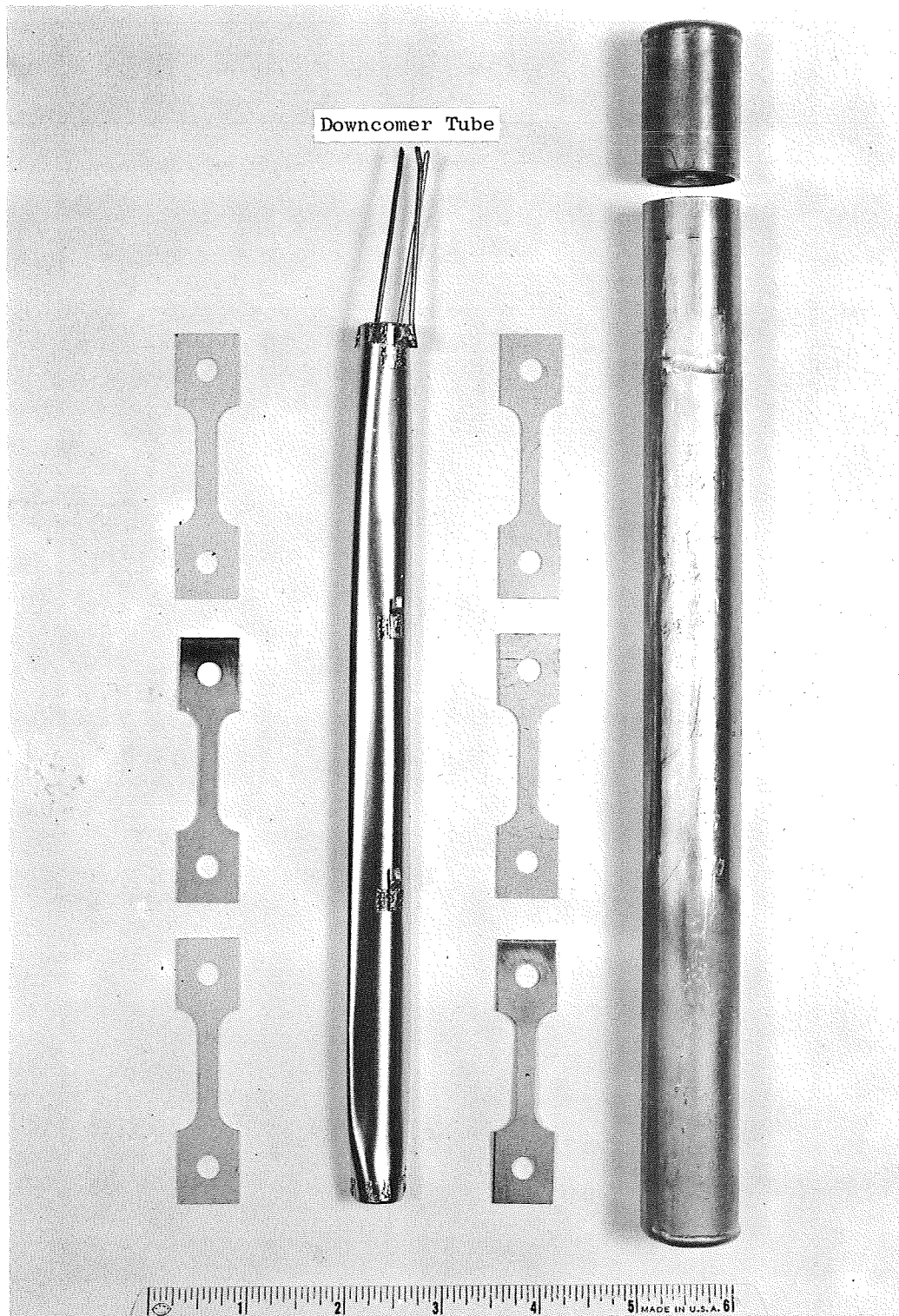


Figure 14. ASTAR 811CN Lithium Thermal Convection Capsule Components Following 5000 Hours Exposure to Flowing Lithium at 2400<sup>o</sup>F.

TABLE IV

WEIGHT MEASUREMENTS OF SPECIMENS EXPOSED TO FLOWING LITHIUM FOR 5000 HOURS  
LITHIUM THERMAL CONVECTION CAPSULE TESTS

Specimen Number	Specimen Condition	Oxygen Concentration, ppm	Specimen Position	Average Temp, °F	Weight Before Test, g	Weight After Test, g	Weight Change, mg
<u>ASTAR 811C Capsule No. 3</u>							
T3	T-111 Oxygen Contaminated <sup>(a)</sup>	442	Bottom Level	2400	14.0189	14.0045	-14.4
T7	T-111 Oxygen Contaminated <sup>(b)</sup>	442	Bottom Level	2400	14.3145	14.2982	-16.3
15E	811C Oxygen Contaminated <sup>(a)</sup>	254	Bottom Level	2400	14.7151	14.7098	- 5.3
16E	811C Oxygen Contaminated <sup>(b)</sup>	254	Bottom Level	2400	14.7945	14.7900	- 4.5
7E	811C Oxygen Contaminated <sup>(a)</sup>	254	Top Level	2100	14.7157	14.7144	- 1.3
8E	811C Oxygen Contaminated <sup>(b)</sup>	254	Top Level	2100	14.7300	14.7295	- 0.5
1F	811C As Received <sup>(a)</sup>	51	Middle Level	2150	14.6978	14.6959	- 1.9
3F	811C As Received <sup>(b)</sup>	51	Middle Level	2150	14.3935	14.3918	- 1.7
R1	811C Rupture Specimen	51	Middle Level	2150	7.9343	7.9368	+ 2.5
R2	811C Rupture Specimen	51	Middle Level	2150	7.3195	7.3193	- 0.2
<u>ASTAR 811CN Capsule No. 5</u>							
1	ASTAR 811CN ↓	89 ↓	Bottom Level	2400	12.6300	12.6266	- 3.4
2			Bottom Level	2400	12.6686	12.6635	- 5.1
3			Middle Level	2150	12.6146	12.6125	- 2.1
4			Middle Level	2150	12.4220	12.4206	- 1.4
5			Top Level	1975	12.5940	12.5881	- 5.9
6			Top Level	1975	12.5216	12.5171	- 4.5

(a) Welded

(b) Welded and postweld annealed at 2400°F/1 hour

TABLE V

BEND TEST RESULTS OF SPECIMENS EXPOSED TO FLOWING LITHIUM FOR 5000 HOURS.  
LITHIUM THERMAL CONVECTION CAPSULE TESTS

PRETEST					POSTTEST				
Specimen Number	Ultimate Load, lbs	Bend Test Results <sup>(a)</sup>	Specimen Condition	Oxygen Concentration ppm	Specimen Number	Specimen Position	Avg. Temp. °F	Ultimate Load, lbs	Bend Test Results <sup>(a)</sup>
T9	87.3	Ductile	T-111 <sup>(b)</sup>	442	T3	Bottom level	2400	78.3	Ductile
T10	91.8	Ductile	T-111 <sup>(c)</sup>	442	T7	Bottom level	2400	81.8	Ductile
1E	125.0	Ductile	ASTAR 811C <sup>(b)</sup>	254	15E	Bottom level	2400	93.0	Ductile
2E	107.6	Ductile	ASTAR 811C <sup>(c)</sup>	254	16E	Bottom level	2400	102.6	Ductile
1E	125.0	Ductile	ASTAR 811C <sup>(b)</sup>	254	7E	Top level	2100	96.0	Slight cracking
2E	107.6	Ductile	ASTAR 811C <sup>(c)</sup>	254	8E	Top level	2100	101.6	Ductile
5F	116.2	Ductile	ASTAR 811C <sup>(b)</sup>	51	1F	Middle level	2150	104.0	Ductile
6F	95.2	Ductile	ASTAR 811C <sup>(c)</sup>	51	3F	Middle level	2150	87.3	Ductile

(a) 1t bend at room temperature to a bend angle of 90° at a strain rate of 0.2 in/sec.

(b) Welded

(c) Welded and postweld annealed at 2400°F/1 hr.

zone cracking evidenced in this specimen is shown in Figure 15. Specimen 7E was not postweld annealed but a duplicate specimen tested under the same conditions (specimen 8E) which was annealed (2400°F/1 hr) remained ductile after exposure to lithium. Specimen 15E which was in the same condition as specimen 7E but tested at an average temperature of 2400°F was ductile.

Also shown in Table V is the fact that lithium exposure reduced the strength of lithium exposed specimens as compared to the strength of the unexposed specimens.

#### d. Stress-Rupture Tests

The six ASTAR 811CN stress-rupture specimens which were exposed to lithium in ASTAR 811CN Capsule No. 5 and two specimens in the pretest condition were stress rupture tested to produce rupture in 100 hours at 2400°F. Difficulty was encountered in the selection of a proper stress which would produce rupture in 100 hours at 2400°F. A stress of 20,000 psi was finally selected and specimens were tested until fracture resulted. The results of the stress-rupture testing are tabulated in Table VI and schematically in Figure 16 showing the respective locations of the samples within the capsule. The results of the stress-rupture testing show that a reduction of strength is observed after exposure to lithium. A comparison of the average rupture life of pretest specimens 7 and 8 tested at 20,000 psi, and the rupture lives of specimens 2, 3 and 6, also tested at 20,000 psi indicates a decrease in stress rupture life as the lithium exposure temperature increases. Based on chemistry results, which will be discussed later, this decrease in strength is associated with a time at temperature relationship rather than to any mass transfer of interstitial elements or significant lithium attack.

#### e. Chemical Analysis

Weld zone and heat affected zone-parent metal sections were cut from the ASTAR 811C and T-111 alloy bend specimens exposed to flowing lithium in ASTAR 811C Capsule No. 3. The specimens were analyzed for oxygen, nitrogen and hydrogen by the vacuum fusion technique and for carbon by the combustion conductometric technique. The analytical



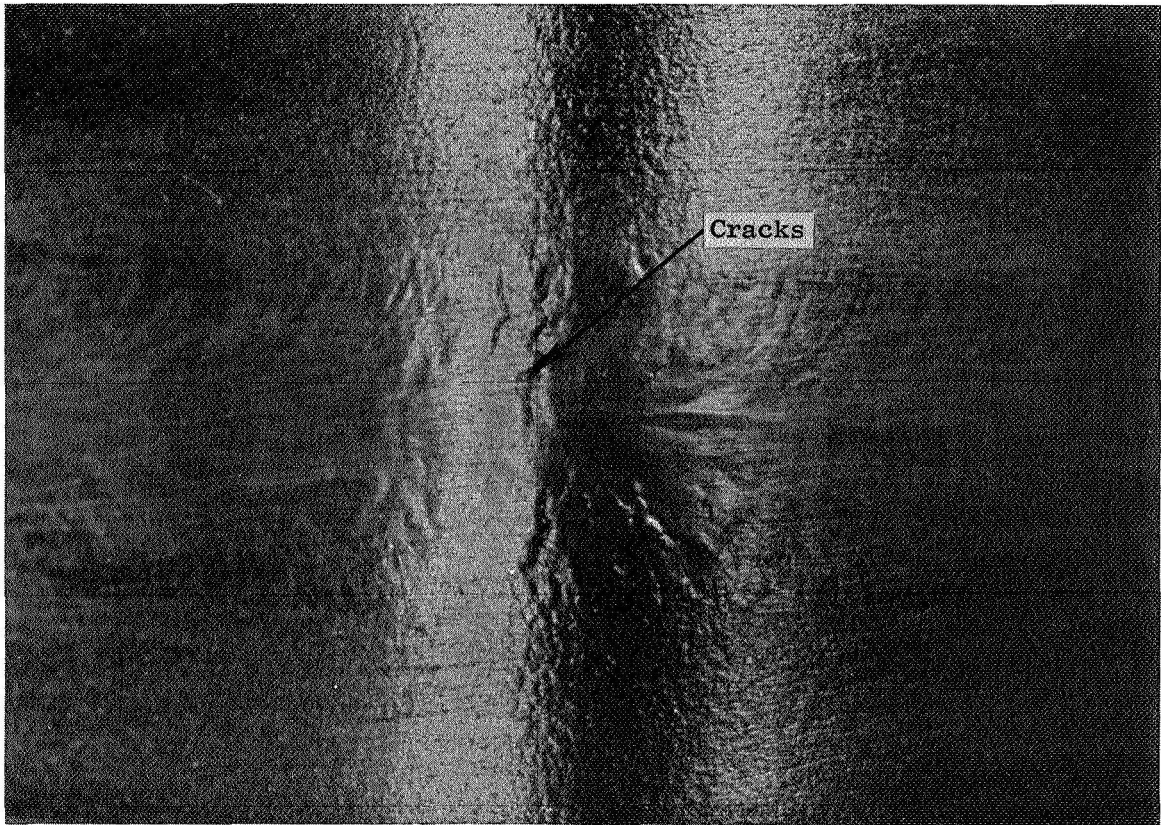


Figure 15. ASTAR 811C Bend Specimen Which Has Been Oxygen Contaminated and Welded Prior to Exposure to Lithium at Approximately 2100°F for 5000 Hours. The Specimen was Bent 90° at lt. Subsequent Metallographic Examination Indicated the Cracks were Associated with Lithium Attack.

TABLE VI

STRESS RUPTURE LIFE OF ASTAR 811CN SPECIMENS BEFORE AND AFTER EXPOSURE TO FLOWING LITHIUM FOR 5000 HOURS  
 ASTAR 811CN LITHIUM THERMAL CONVECTION CAPSULE NO. 5

Specimen Number	Condition	Vacuum Level ( $\times 10^{-6}$ Torr)	Stress <sup>(a)</sup> (ksi)	Rupture Life <sup>(b)</sup> (hrs)	Elong. (%)	Specimen Position	Average Lithium Temperature, °F
1	posttest	2.4	25.0	3.7	51.0	Bottom level	2400
2	posttest	1.4	20.0	50.3	47.0	Bottom level	2400
3	posttest	0.8	20.0	100.8	38.0	Middle level	2150
4	posttest	1.0	20.0	5.3	50.0	Middle level	2150
5	posttest	2.0	13.0	100.2 (R)	0.7	Top level	1975
5	posttest	5.0	20.0	247.4	49.0	Top level	1975
6	posttest	2.4	20.0	120.2	43.0	Top level	1975
7	pretest	3.8	13.0	100.5 (R)	0.5	-	-
7	pretest	3.6	20.0	158.3	34.0	-	-
8	pretest	2.0	13.0	200.1 (R)	0.8	-	-
8	pretest	7.2	20.0	171.3	32.0	-	-

(a) All tests performed at 2400°F

(b) (R) indicates removal at time specified

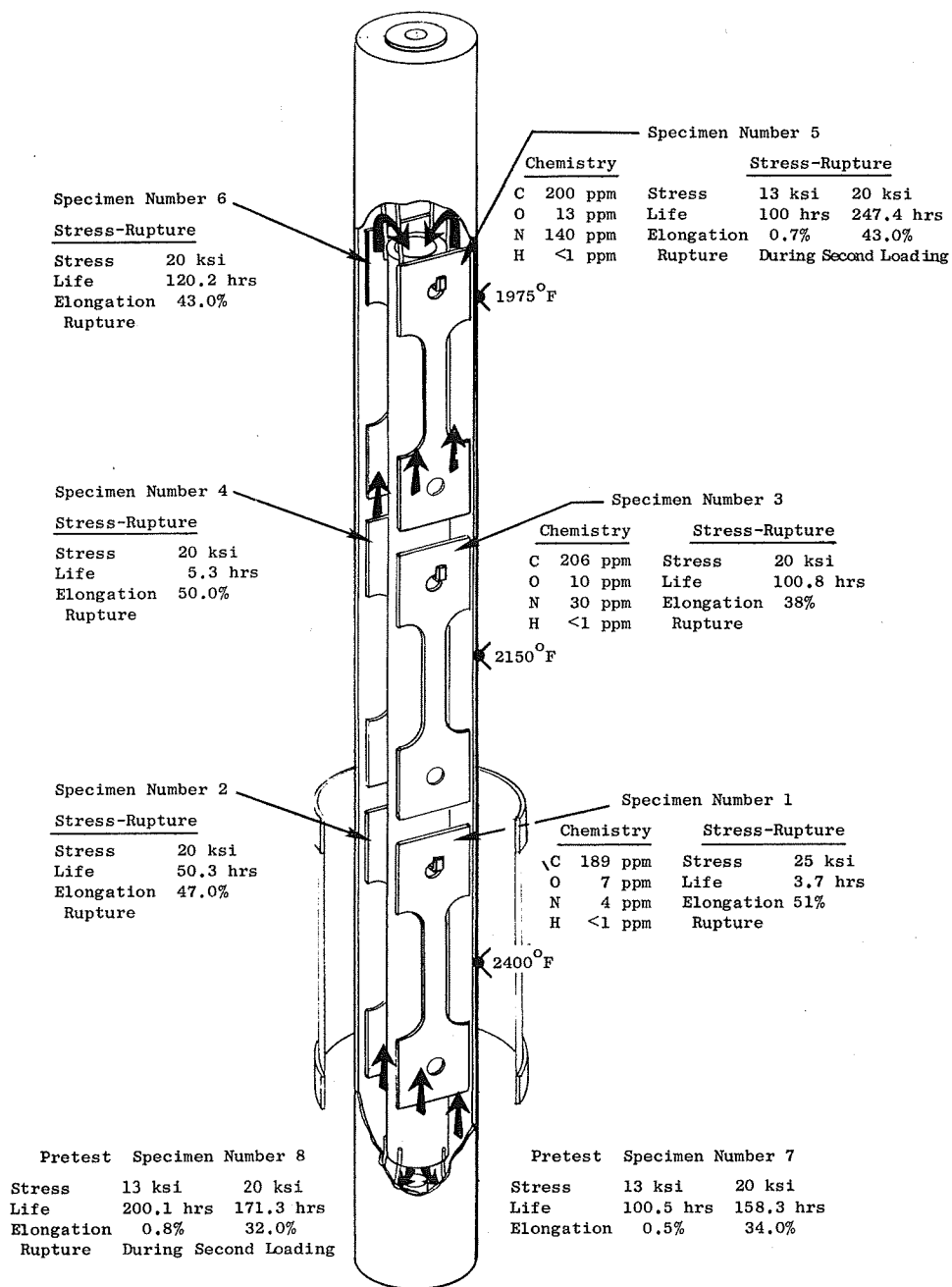


Figure 16. Stress-Rupture and Chemical Analysis Results of Specimens in ASTAR 811CN Capsule No. 5 After 5000 Hours Exposure to Flowing Lithium. Stress-Rupture Tests were Performed at 2400°F.

results are presented in Table VII and the averaged results are shown schematically in Figure 17 to indicate the position of the specimen during testing. The oxygen concentration of all specimens was reduced to 10 ppm or less regardless of whether the specimens were attacked by lithium. This oxygen dissolution effect has been observed in other refractory metal-alkali metal systems.<sup>(3)</sup> The mass transfer of nitrogen from the hottest specimens at the bottom level of the capsule to those at the top is evident whereas the changes in carbon concentration are more subtle.

Very similar analytical results shown in Table VIII were obtained on ASTAR 811CN stress rupture specimens exposed in ASTAR 811CN Capsule No. 5. Only the results obtained on the specimens before stress-rupture testing should be considered in explaining the effects of lithium exposure. The analysis of the tested stress rupture specimens is influenced by contamination of the specimens during stress rupture testing, although all the tests were performed in vacuums in the  $10^{-6}$  torr range. Stress rupture testing at this vacuum level must not exceed 100 hours to prevent contamination which could effect the accuracy of the test results. The specimens with oxygen concentrations over 100 ppm were all tested over 100 hours. As an example, ASTAR 811CN Specimen 8, with an oxygen concentration of 276 ppm, was tested for 370 hours (Table VI).

Chemical analysis was obtained on capsule wall segments adjacent to the specimens. The results for the ASTAR 811C capsule are presented in Table IX. Mass transfer of carbon as well as nitrogen and oxygen from the hottest portion of the capsule to the cooler end is more evident in the analytical results for the capsule material than previously described results for the specimens suspended in the capsule. Chemistry changes in the capsule wall are of more interest since the capsule wall better simulates lithium containment piping in a space power system.

The chemical analysis of ASTAR 811CN capsule wall material, the results of which are shown in Table X, are somewhat different from those observed on the ASTAR 811C capsule. Oxygen and nitrogen mass transfer

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(3) Harrison, R. W., "Corrosion of Oxygen Contaminated Tantalum in NaK," Topical Report 1, NASA Contract NAS 3-10610, January 1969, GESP-138.

TABLE VII

CHEMICAL ANALYSES OF ASTAR 811C AND T-111 ALLOY SPECIMENS BEFORE AND AFTER  
EXPOSURE TO FLOWING LITHIUM FOR 5000 HOURS IN ASTAR 811C CAPSULE 3

Specimen Number	Average Temp. °F	Specimen Condition	Chemical Analyses Location	Concentration, ppm			
				C	O	N	H
T9	-	T-111 oxygen contaminated <sup>(a)</sup>	Weld nugget	50,54	351,343	9,8	< 1
		Pretest	HAZ - PM <sup>(c)</sup>	66,59	387,382	11,6	< 1
T3	2400	T-111 oxygen contaminated <sup>(a)</sup>	Weld nugget	84,57	8,8	3,3	1,1
		Posttest	HAZ - PM	38,41	6,4	3,3	< 1
T7	2400	T-111 oxygen contaminated <sup>(b)</sup>	Weld nugget	35,34	2,5	< 1,5	< 1
		Posttest	HAZ - PM	23,25	3,4	4,4	< 1
1E	-	811C oxygen contaminated <sup>(a)</sup>	Weld nugget	274,284	184,179	6,6	< 1
		Pretest	HAZ - PM	330,324	233,248	16,7	< 1
15E	2400	811C oxygen contaminated <sup>(a)</sup>	Weld nugget	197,199	9,16	1,1	< 1
		Posttest	HAZ - PM	284,272	9,7	2,1	< 1
16E	2400	811C oxygen contaminated <sup>(b)</sup>	Weld nugget	192,212	10,6	1, < 1	< 1
		Posttest	HAZ - PM	268,283	6,16	1,1	< 1
7E	2100	811C oxygen contaminated <sup>(a)</sup>	Weld nugget	227,247	11,11	15,12	1,1
		Posttest	HAZ - PM	285,268	8,3	40,36	< 1
8E	2100	811C oxygen contaminated <sup>(b)</sup>	Weld nugget	243,205	6,7	28,60	< 1
		Posttest	HAZ - PM	268,272	11,3	55,47	< 1
	-	811C noncontaminated	Not welded	290,332	43,59	9,8	1,1
		Pretest					
1F	2150	811C noncontaminated <sup>(a)</sup>	Weld nugget	260,261	5,4	21,11	< 1
		Posttest	HAZ - PM	303,313	1,2	12,15	< 1
3F	2150	811C noncontaminated <sup>(b)</sup>	Weld nugget	245,241	3,1	9,6	< 1
		Posttest	HAZ - PM	285,306	< 1	9,10	< 1

(a) Welded

(b) Welded and postweld annealed at 2400°F/1 hr

(c) Heat affected zone - Parent metal

(d) Duplicate analysis

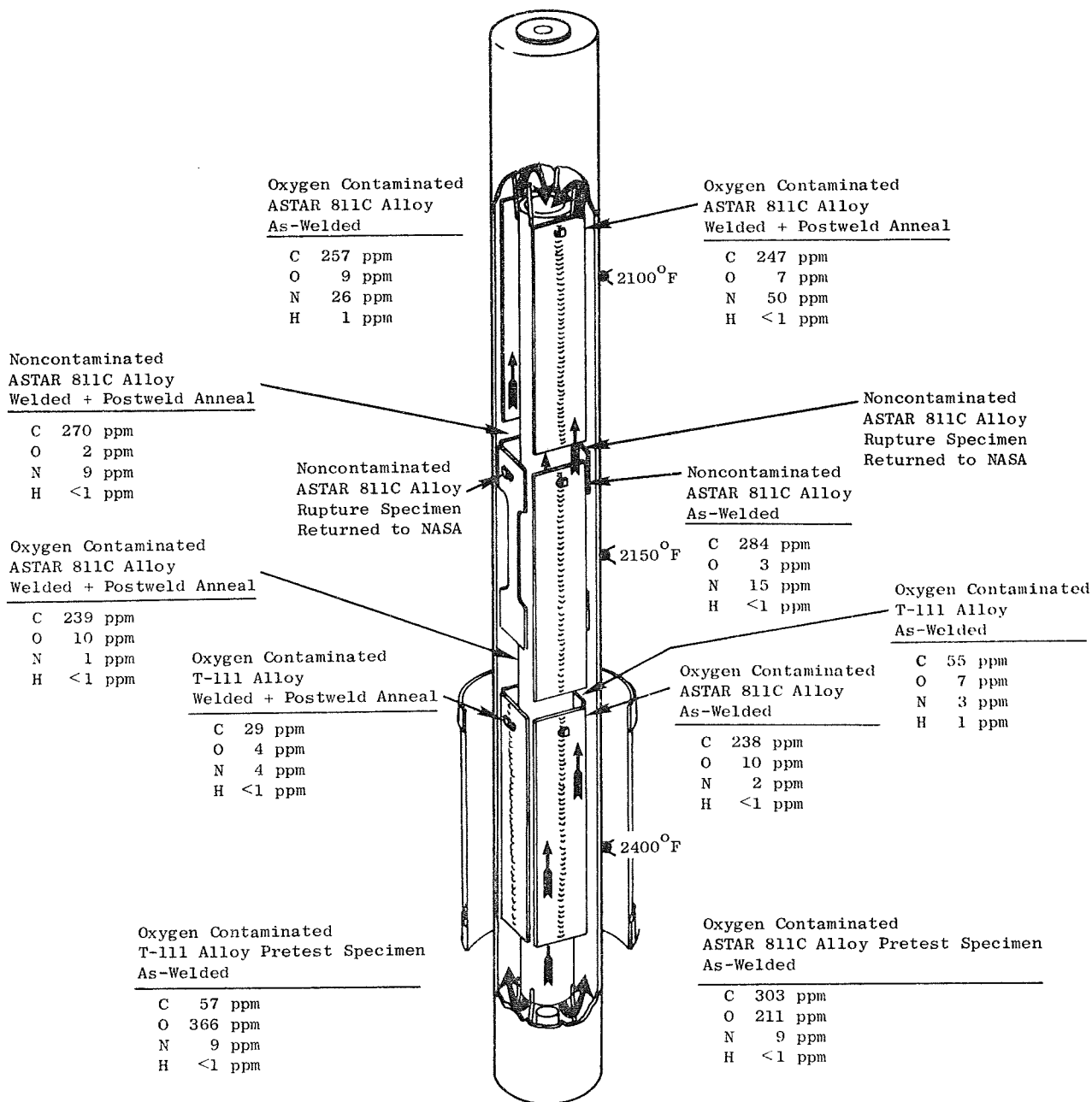


Figure 17. Chemical Analysis Results of Specimens in ASTAR 811C Capsule No. 3 After 5000 Hours Exposure to Flowing Lithium.

TABLE VIII

CHEMICAL ANALYSES OF ASTAR 811CN SPECIMENS EXPOSED TO FLOWING LITHIUM FOR 5000 HOURS  
ASTAR 811CN LITHIUM THERMAL CONVECTION CAPSULE NO. 5

<u>BEFORE STRESS RUPTURE TESTING</u>										
Specimen Number	Average Temp. °F	Specimen Location	Concentration, ppm							
			Duplicate Analyses				Average Analyses			
			C	O	N	H	C	O	N	H
1	2400	Bottom	189	5,8	3,4	< 1	189	7	4	< 1
3	2150	Middle	206	12,8	42,12	< 1	206	10	30	< 1
5	1975	Top	200	14,11	164,115	< 1	200	13	140	< 1
	-	Pretest					163	89	107	< 1
<u>AFTER STRESS RUPTURE TESTING</u> <sup>(b)</sup>										
Specimen Number	Average Temp. °F	Specimen Location	S.R. Test Hours <sup>(a)</sup>	Concentration, ppm						
				C	O	N	H	C	O	N
1	2400	Bottom	3.7	136,173	33,46	2,3	1,2	155	40	3
2	2400	Bottom	50.3	148,169	52,60	2,4	< 1	159	56	3
3	2150	Middle	100.8	219,230	57,79	49,44	< 1	225	68	47
4	2150	Middle	5.3	181,191	46,55	48,60	< 1	186	51	54
5	1975	Top	347.6	340,382	196,165	156,140	< 1	361	181	148
6	1975	Top	120.2	152,190	82,126	178,108	< 1	171	104	143
7	-	Pretest	258.8	278,253	198,218	99,89	< 1	266	208	94
8	-	Pretest	371.4	238,231	260,292	97,84	< 1	235	276	91

(a) Total time at stress rupture temperature

(b) All stress-rupture tests performed at 2400°F

TABLE IX

CHEMICAL ANALYSES OF ASTAR 811C ALLOY CAPSULE WALL MATERIAL  
AFTER 5000 HOURS EXPOSURE TO FLOWING LITHIUM  
LITHIUM THERMAL CONVECTION CAPSULE NO. 3

Specimen Location	Average Temp, °F	Concentration, ppm							
		Duplicate Analyses				Average Analyses			
		C	O	N	H	C	O	N	H
Pretest Material		290,332	43,59	9,8	1,1	311	51	9	1
Posttest - Lower Level	2400	203,230	6,6	5,7	< 1	217	6	6	< 1
Posttest - Middle Level	2150	225,269	71,67	14,14	< 1	247	69	14	< 1
Posttest - Upper Level	2100	303,290	119,129	56,62	1,1	297	124	59	1



TABLE X

CHEMICAL ANALYSES OF ASTAR 811CN ALLOY CAPSULE WALL MATERIAL  
AFTER 5000 HOURS OF EXPOSURE TO FLOWING LITHIUM  
LITHIUM THERMAL CONVECTION CAPSULE NO. 5

Specimen Location	Average Temp, °F	Concentration, ppm							
		Duplicate Analyses				Average Analyses			
		C	O	N	H	C	O	N	H
Pretest Material		152,174	91,87	113,101	< 1	163	89	107	< 1
Posttest - Lower Level	2400	199,182	5,3	3,4	1,1	191	4	4	1
Posttest - Middle Level	2150	180,183	50,49	58,68	1,1	182	50	63	1
Posttest - Upper Level	1975	169,160	129,113	151,124	< 1	165	121	136	< 1

are similar but lesser in extent whereas mass transfer of carbon from the hot to cold end of the capsule is not evidenced. It is believed that the observed differences can be attributed to the lithium flow in the ASTAR 811CN capsule which was obscured by the position of the downcomer tube on the bottom of the capsule. Flow in this capsule was observed to be intermittent in nature, swirling up and down the annular space between the capsule and downcomer tube. This difference in flow between the capsules is further described in the temperature differences observed between the hot and cold end of the capsules. The  $\Delta T$  observed in the ASTAR 811C capsule with flow occurring as indicated in the capsule schematic in Figure 17 was 300°F as compared to the  $\Delta T$  in the ASTAR 811CN capsule with limited lithium flow, 425°F. It is therefore projected that it is necessary to conduct tests in flowing alkali metals to ascertain the stability of interstitial strengthened alloys.

#### f. Metallographic Examination

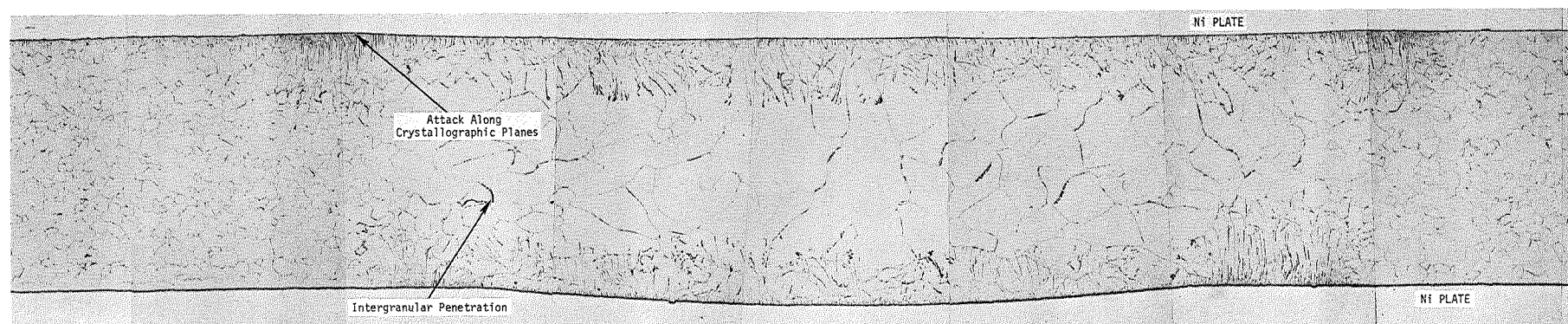
Transverse sections of ASTAR 811C and T-111 alloy bend specimens from ASTAR 811C Capsule No. 5 were examined to determine the effects of the lithium exposure on the microstructure of each alloy as a function of oxygen concentration and specimen condition i.e. welded, welded and postweld annealed.

The most extensive lithium attack was observed in the ASTAR 811C specimens which had been oxygen contaminated and exposed in the as-welded condition. The microstructure of such an ASTAR 811C specimen is compared with that of a T-111 specimen in the same condition in Figure 18. Both specimens were exposed to flowing lithium at an average temperature of 2400°F in the bottom level of ASTAR 811C Capsule No. 3. The lithium attack in the ASTAR 811C specimen is greatest in the heat affected zone adjacent to the weld. Platelet type attack across specific crystallographic planes as well as intergranular penetration was observed. This type of corrosion is typical in refractory alloys exposed to lithium when sufficient oxygen is in solution in the metal to combine with the lithium resulting in attack.<sup>(4,5)</sup> The observed attack along crystal-

(4) Harrison, R. W., "The Effects of Welding Atmosphere Purity on the Lithium Corrosion Resistance of Refractory Alloys," presented at the Symposium on Corrosion by Liquid Metals, Philadelphia, Pa. October 13, 1969, proceedings to be published by Plenum Press.

(5) Harrison, R. W., and Hoffman, E. E., "The Compatibility of Refractory Metals with Liquid Metals," Refractory Metal Alloy Metallurgy and Technology, Plenum Press, N.Y., 1968, p. 251.

ASTAR 811C + 211 PPM OXYGEN



T-111 + 366 PPM OXYGEN

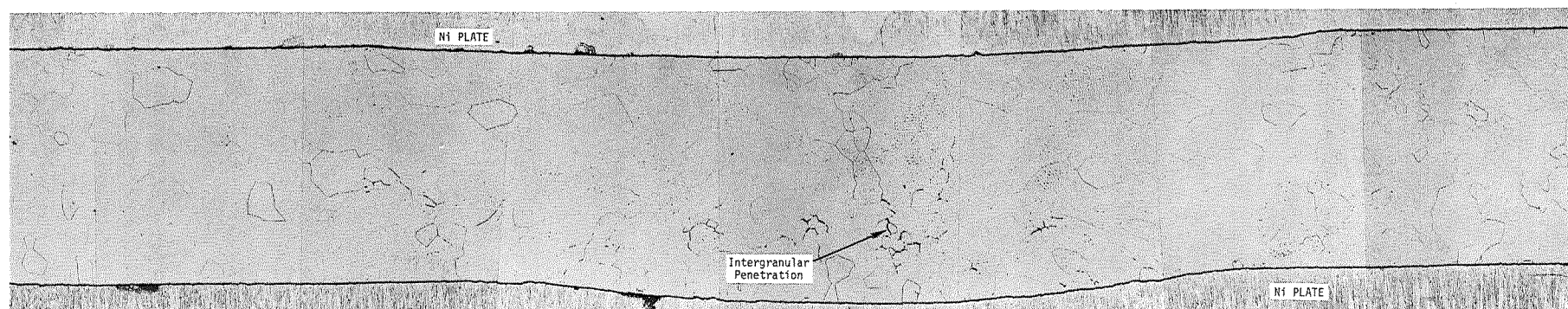


Figure 18. Corrosion in Welded ASTAR 811C and T-111 Oxygen Contaminated Specimens Following 5000 Hour Exposure to Flowing Lithium at 2400°F.

Etchant: 30g  $\text{NH}_4\text{F}$  - 20 ml  $\text{HNO}_3$  - 50 ml  $\text{H}_2\text{O}$

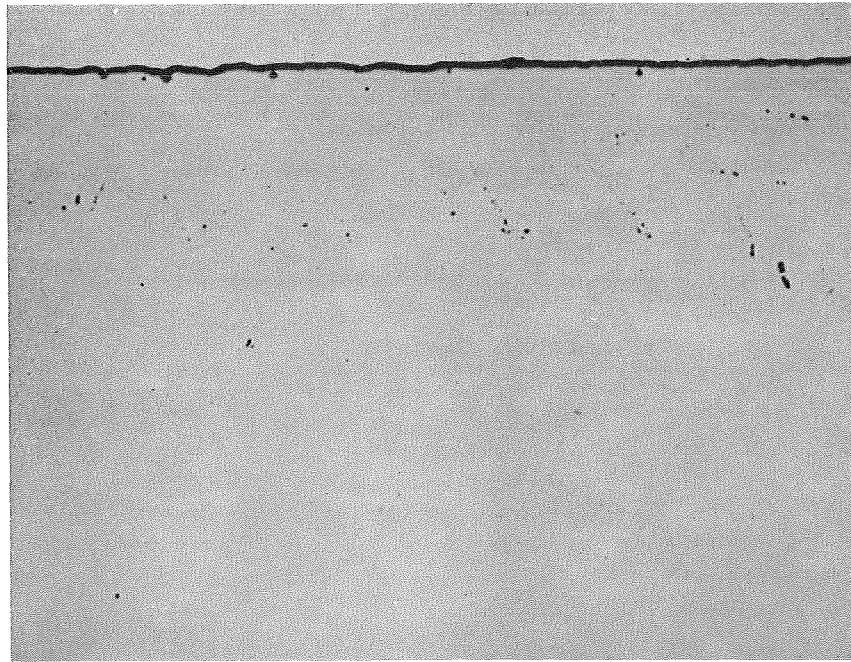
75X

lographic planes, which are believed to be rich in oxygen, extends to a depth of 15 mils whereas intergranular corrosion extends throughout the entire specimen thickness. In comparison, attack in the T-111 alloy specimen was observed in the weld zone only and was much less extensive. Considering that the oxygen concentration in the T-111 alloy specimen before exposure was much greater than that of the ASTAR 811C specimen the differences in observed attack are surprising. Undoubtedly the differences can be attributed to the differences in the hafnium concentrations in these alloys, 0.7% Hf in the ASTAR 811C alloy and 2% Hf in the T-111 alloy. The relationship between the amount of getter alloy additions to refractory metals and their susceptibility to lithium attack has been well established.<sup>(6)</sup> The smaller hafnium addition in the ASTAR 811C alloy while having considerable advantages in improving the high temperature creep strength of the alloy has considerable disadvantages in reducing the alloys corrosion resistance to lithium. A more dramatic indication of this was observed in the uncontaminated ASTAR 811C specimen exposed to flowing lithium in the as-welded condition. The oxygen concentration of this specimen before exposure was only 50 ppm. Posttest metallographic examination of this specimen indicated lithium attack in the heat affected zone adjacent to the weld of 6 mils depth as shown in Figure 19. Corrosion in a welded refractory alloy containing such a small amount of oxygen imposes considerable restraints on system fabrication.

Preliminary metallographic evidence has indicated postweld annealing (1 hour at 2400°F) of welded specimens has eliminated lithium attack. The ability of postweld annealing to prevent attack is directly related to the amount of oxygen that will combine with the getter alloy addition in refractory alloys. Stoichiometry indicates the following:

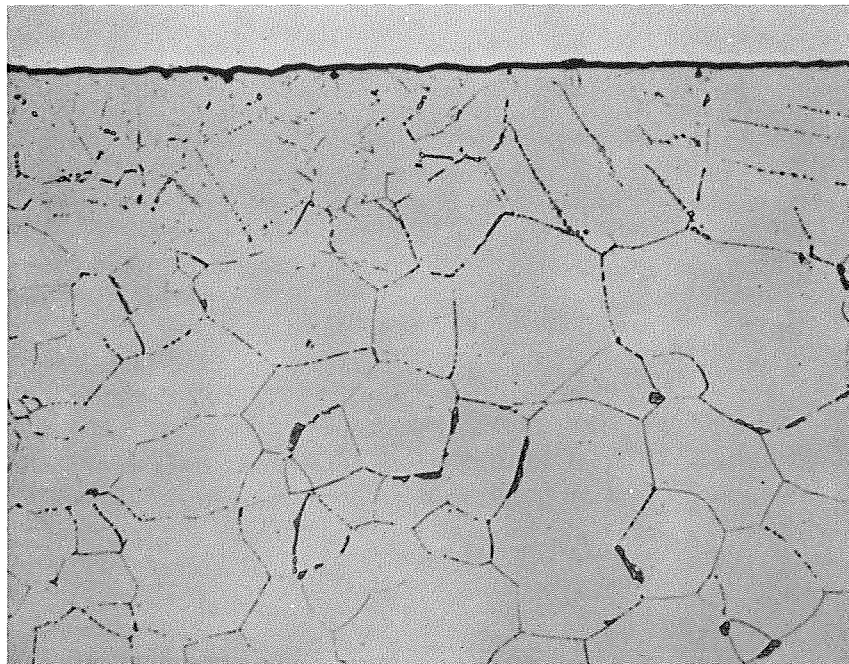
Alloy	Maximum Oxygen Concentration Possible Combined as ZrO <sub>2</sub> or HfO <sub>2</sub>	
	ppm	Atomic (%)
Cb-1Zr (1% Zr)	3500	2.02
T-111 (2% Hf)	3580	3.97
ASTAR 811C (0.7% Hf)	1250	1.38
ASTAR 811CN (1% Hf)	1790	1.98

(6) DiStefano, J. R. and Litman, A. P., "Effects of Impurities in Some Refractory Metal-Alkali Metal Systems," Corrosion, December 1964, p. 392t.



↑  
Depth  
of  
Attack  
6 mils  
↓

As-Polished



↑  
Depth  
of  
Attack  
6 mils  
↓

Etched  $30\text{NH}_4\text{F}-20\text{HNO}_3-50\text{H}_2\text{O}$

Figure 19. Corrosion in Welded ASTAR 811C Containing 50 ppm Oxygen Prior to Exposure to Flowing Lithium for 5000 Hours at 2100° F.

Based on Cb-1Zr technology, oxygen concentrations in considerable excess of the above levels would render these alloys susceptible to lithium attack regardless of the heat treatment.

#### IV. FUTURE PLANS

- A. Continue operation of the T-111 Rankine System Corrosion Test Loop.
- B. Complete evaluation of the ASTAR alloy specimens from the lithium thermal convection capsules.
- C. Continue testing the refluxing potassium capsule tests of ASTAR 811C alloy.
- D. Complete fabrication and instrumentation of the 1900°F Lithium Loop.





## V. APPENDIX

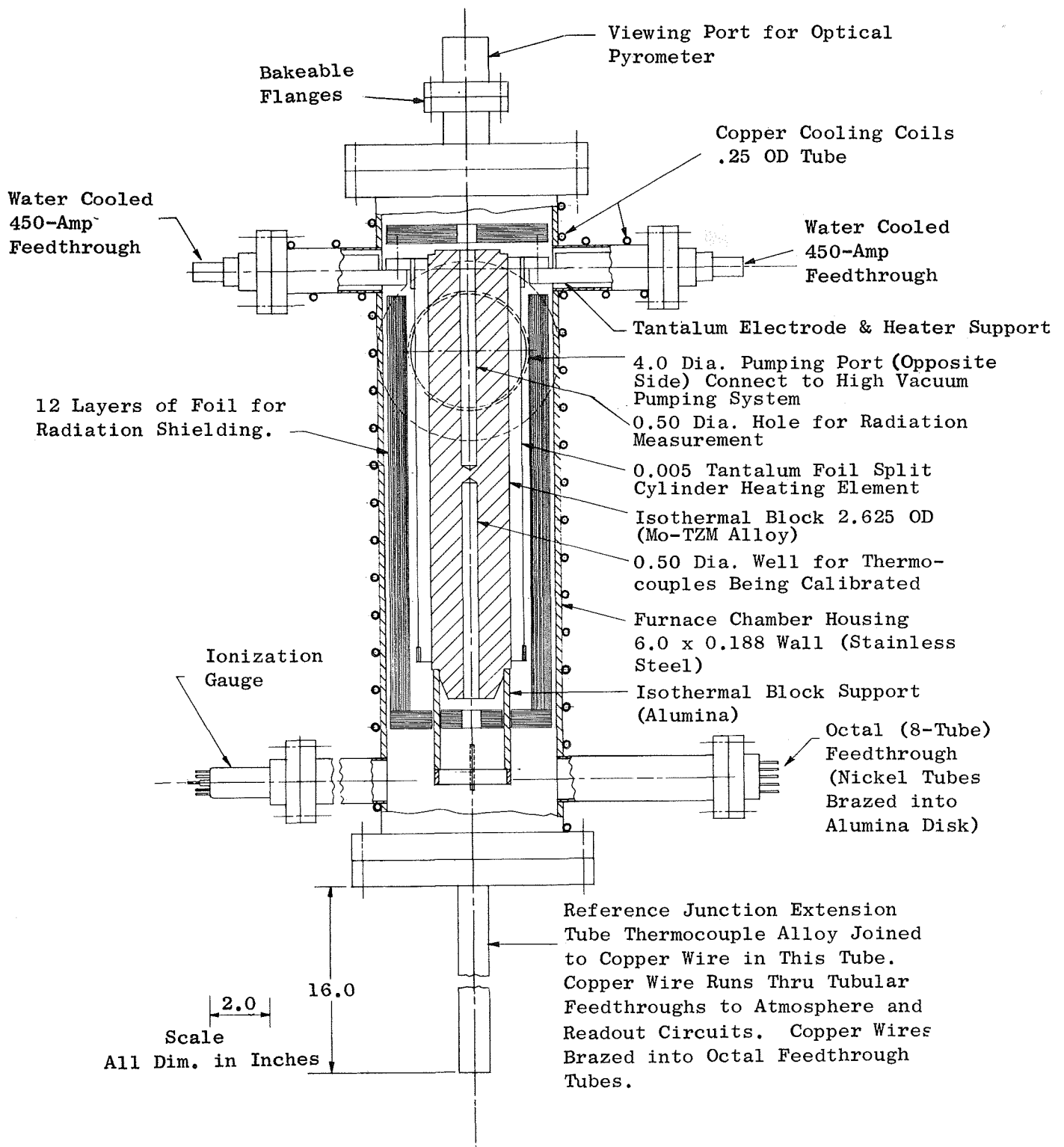
CALIBRATION OF W-3Re/W-25Re THERMOCOUPLE WIRE

Calibration of the thermocouple wire used on the T-111 Rankine System Corrosion Test Loop was conducted in a high-vacuum environment with sample thermocouples made from the same spools of wire which were used to instrument the loop. This is the same wire used to instrument the Cb-1Zr Rankine System Corrosion Test Loop. A complete description of the apparatus used, procedures, and test results has been previously reported.<sup>(1)</sup>

The calibration was conducted in a vacuum furnace chamber constructed especially for the purpose. A sketch of this unit is shown in Figure 20. It consists of a 6-inch-OD tube approximately 2 feet in length with appropriate high-vacuum flanges to provide access to either end. A total of seven 1.5-inch ports were provided for power and thermocouple lead feedthroughs. An isothermal block, 2.6-inch OD and 15 inches long and made of Mo-TZM alloy, was mounted vertically in the center of the chamber. Various components of the calibration system are shown in Figure 21. Thermocouples to be calibrated were installed in a 0.5-inch hole drilled along the axis of the block approximately 7 inches deep from the lower end. A similar hole was provided from the top end of the block with a 0.25-inch web between the bottoms of the two holes. The upper hole was used for optical pyrometer readings of block temperature. Standard reference Pt/Pt-10Rh thermocouples were installed in the bottom hole together with the W-3Re/W-25Re thermocouples to be calibrated. The optical pyrometer and the Pt/Pt-10Rh thermocouples were used as independent means of measuring isothermal block temperatures.

The isothermal block was heated by means of a split cylindrical resistance element made from 0.005-inch-thick tantalum foil. The element was supported on the 450-amp feedthroughs so that it was concentric with

(1) E. E. Hoffman and J. Holowach, Potassium Corrosion Test Loop Development Topical Report No. 7 (Contract NAS 3-2547) GESP-246, R67SD3016 (May, 1968).



**Figure 20.** Vacuum Furnace for Calibrating W-3Re/W-25Re Thermocouple Wire.

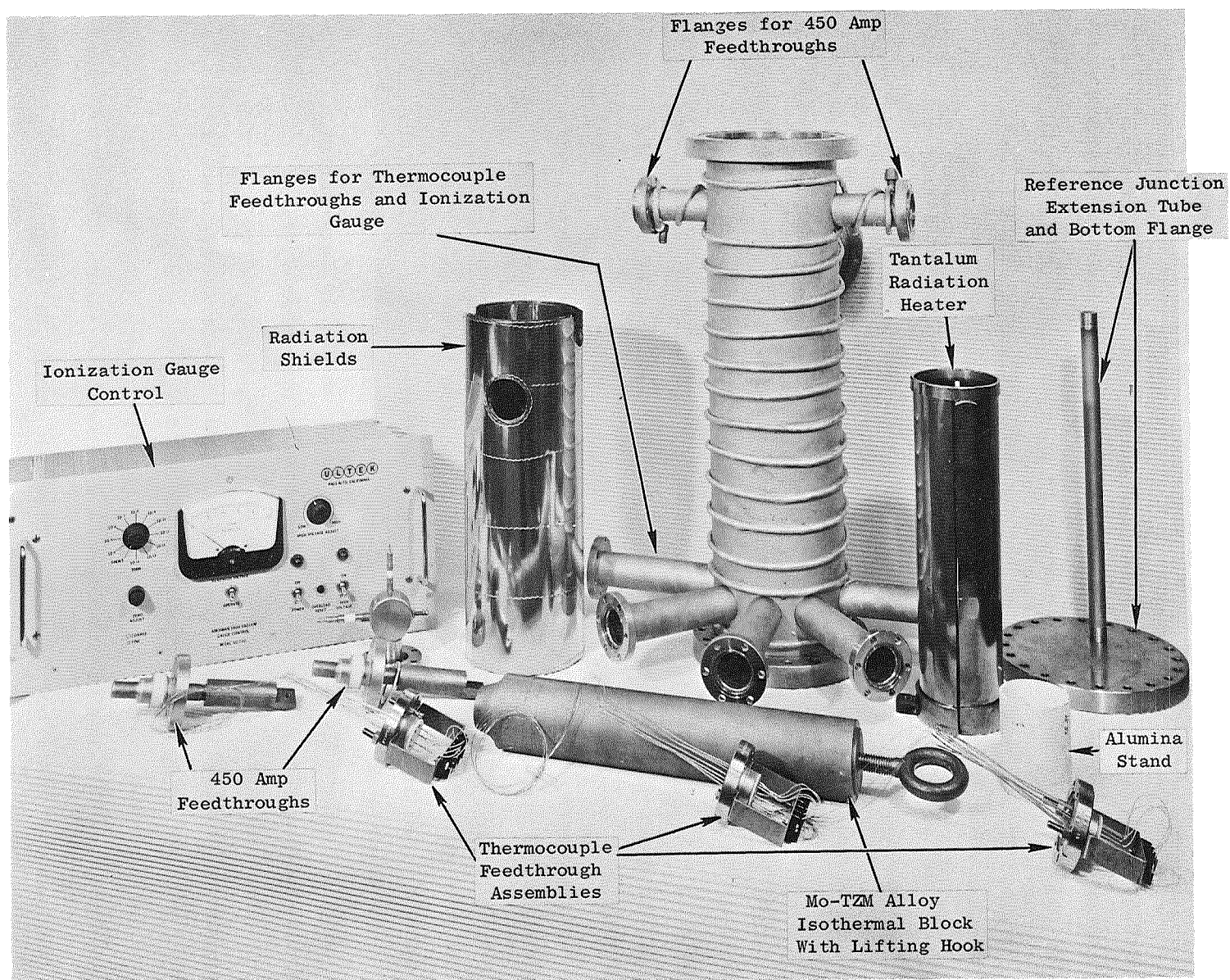


Figure 21. Components of Thermocouple Calibration Vacuum Furnace Before Assembly.  
(C65012206)

the isothermal block. Cylindrical radiation shields were provided to minimize the heat losses from the element to the walls of the chamber. A total of 12 shields were constructed of 0.005-inch-thick foil separated by 0.020-inch tantalum wire. The furnace chamber was attached to the bell jar of a Varian vacuum system containing a 1000-liter/second ion pump as shown in Figure 22. Also shown in this picture are the switches and readout instrument used during the calibration. The readout instrument was a Honeywell Precision Indicator with an accuracy of  $\pm 0.02$  percent of any individual millivolt span from 1 to 70 with a 3-microvolt dead band.

Thermocouples to be calibrated were arranged as shown in Figure 23. Two Pt/Pt-10Rh reference thermocouples made of 0.020-inch-diameter wire were strung through a 4-hole 99 percent purity alumina insulator (0.187-inch OD). A piece of Cb-lZr tubing (0.250-inch OD) was drilled out to make a tight fit and was slipped over the 4-hole insulator containing the Pt/Pt-10Rh reference thermocouples. Samples of the W-3Re and W-25Re wire to be calibrated were strung through 2-hole 99 percent alumina insulators (0.062-inch OD) 30 inches long, and a 1-inch length of 2-hole 0.032-inch 99.5 percent BeO was added to eliminate contact and possible reaction between Cb-lZr and alumina. Four such thermocouples were made from consecutive lengths of wire to be used for instrumenting the loop. These individual thermocouple assemblies with four other samples of different wire were attached to the insulator containing the reference thermocouples by means of Cb-lZr foil straps. The entire assembly was made rigid to eliminate the possibility of slippage between various parts. Three of the hot junctions were formed by resistance welding the individual alloy wires to the Cb-lZr tube in a manner similar to that to be used in the actual loop application. One hot junction was formed by twisting the alloy wires together with no spot weld. Both types of junctions are evident in Figure 23.

The assembly described above was inserted in the calibration furnace as a single unit and strapped down to the chamber structure so that none of the thermocouple wires was in contact with the isothermal block. Copper leads from the vacuum feedthroughs were joined to the alloy wires by twisting both wires together. A copper-constantan thermo-

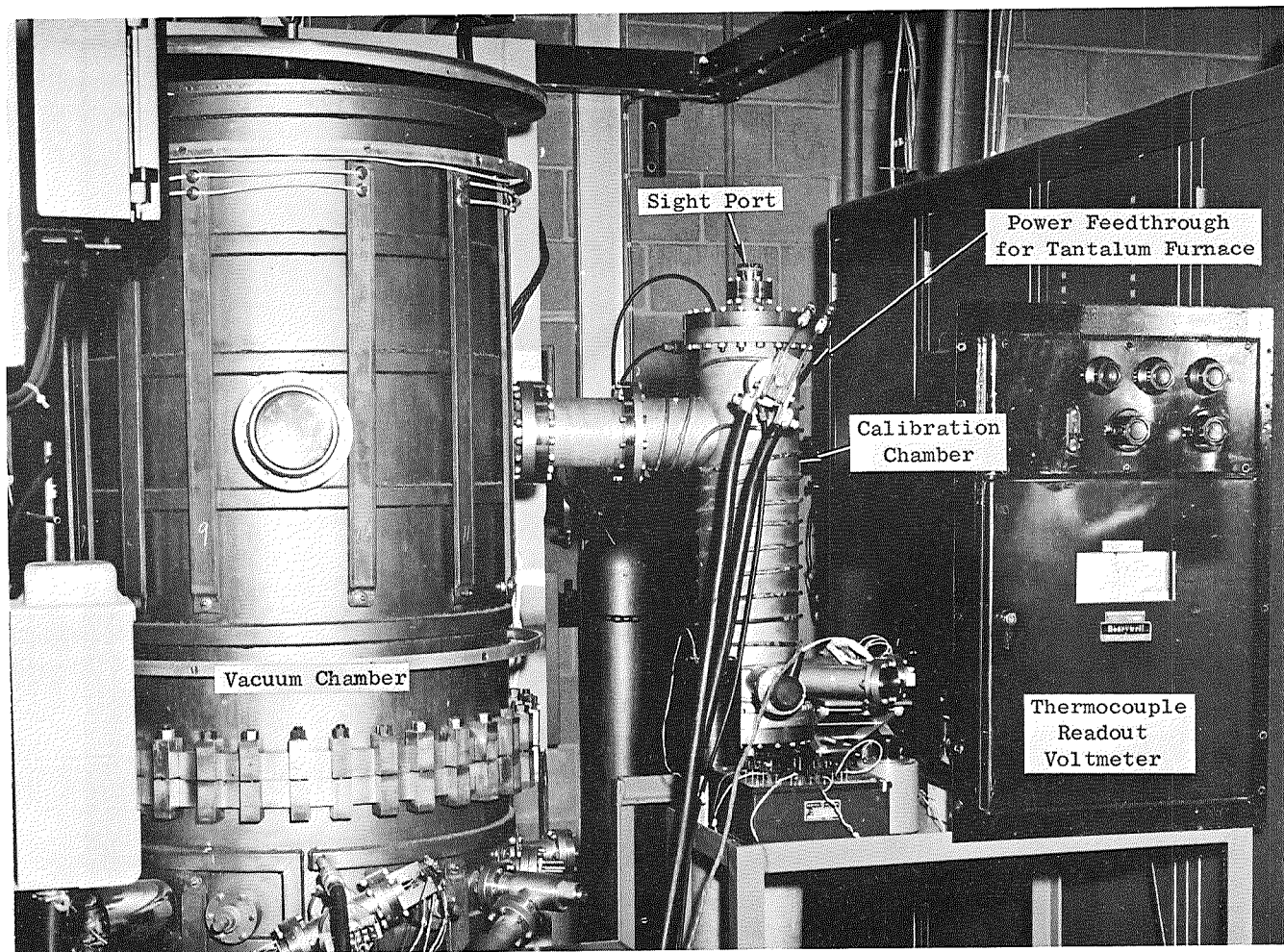


Figure 22. Thermocouple Calibration Furnace Attached to the 24-Inch Diameter Getter-Ion Pumped Vacuum Chamber. (Orig. C65040528)



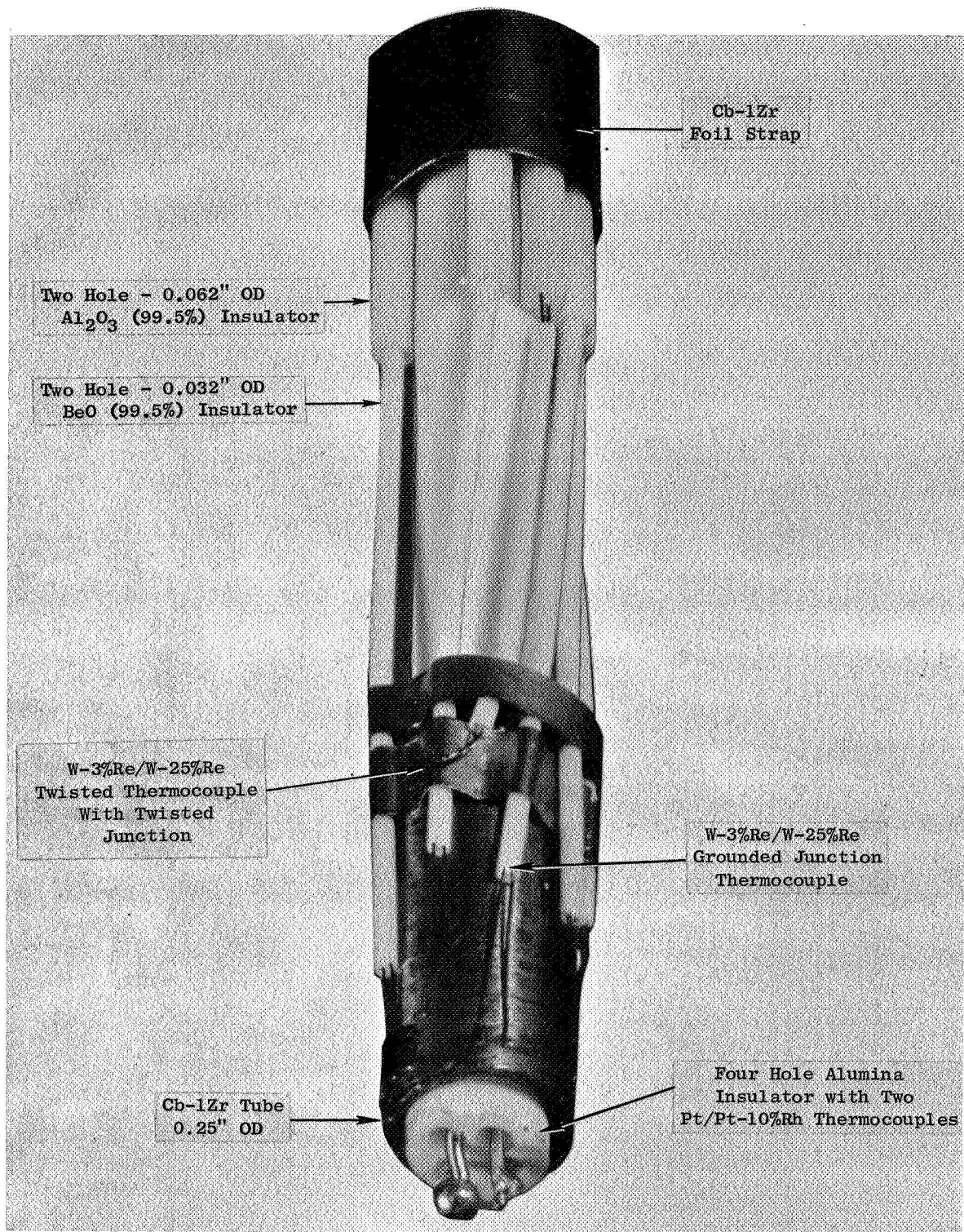


Figure 23. Thermocouple Bundle Used in Calibration of W-3Re/W-25Re Thermocouple Wire. (Orig. C65031521)

couple junction was located in the extension tube to measure the actual cold junction temperature. Calibration data were taken during three temperature cycles. During these runs, the pressure in the calibration furnace varied between a low of  $2 \times 10^{-8}$  torr at 400°F and a high of  $6 \times 10^{-6}$  torr at 2400°F.

Data were reduced by the following procedure.

Step 1. Cold junction temperature was converted from millivolts (copper-constantan thermocouple) to degrees Fahrenheit.

Step 2. Block temperature was determined by adding an emf equivalent to the cold junction temperature to the reference thermocouple output and converting the sum to degrees Fahrenheit by means of National Bureau of Standards Bulletin 561. An average of the two Pt/Pt-10Rh thermocouple readings (never more than 2.8°F of difference) was used as the actual block temperature.

Step 3. A table of output values for each thermocouple being calibrated was prepared from test data in the range 32°-140°F.

Step 4. The millivolt signal equivalent to the reference junction temperature as defined in Step 1 above was calculated for each thermocouple being calibrated by linear interpolation of the table defined as Step 3.

Step 5. A corrected output signal was calculated by adding the millivolt signal determined in Step 4 above to the measured value. This information was used to generate a characteristic curve of corrected output millivolts vs the actual block temperature determined in Step 2 above.

Step 6. In order to facilitate curve plotting and permit use of an expanded scale, a straight line defined by coordinates (0,0) and (24, 2400) where the first number is corrected millivolt output signal, and the second is temperature in °F was used as a base line, and the difference between this line and the corrected output millivolts was calculated. The

difference is plotted vs temperature in Figure 24.

The curve shown in Figure 24 is an average of all data points taken during three temperature cycles over the range from 80° to 2400°F for two thermocouples with different hot junction configurations but from the same spools of alloy wire. All but 8 of 35 stable points fall within 4°F of this line. Maximum deviation of any stable point from the line (up to 2200°F) is 9°F. The actual values used to define the curve are listed in Table XI. These are not actual test points but were picked off of the best line through the test data so that linear interpolation between any two points would cause a maximum error of 0.5°F.



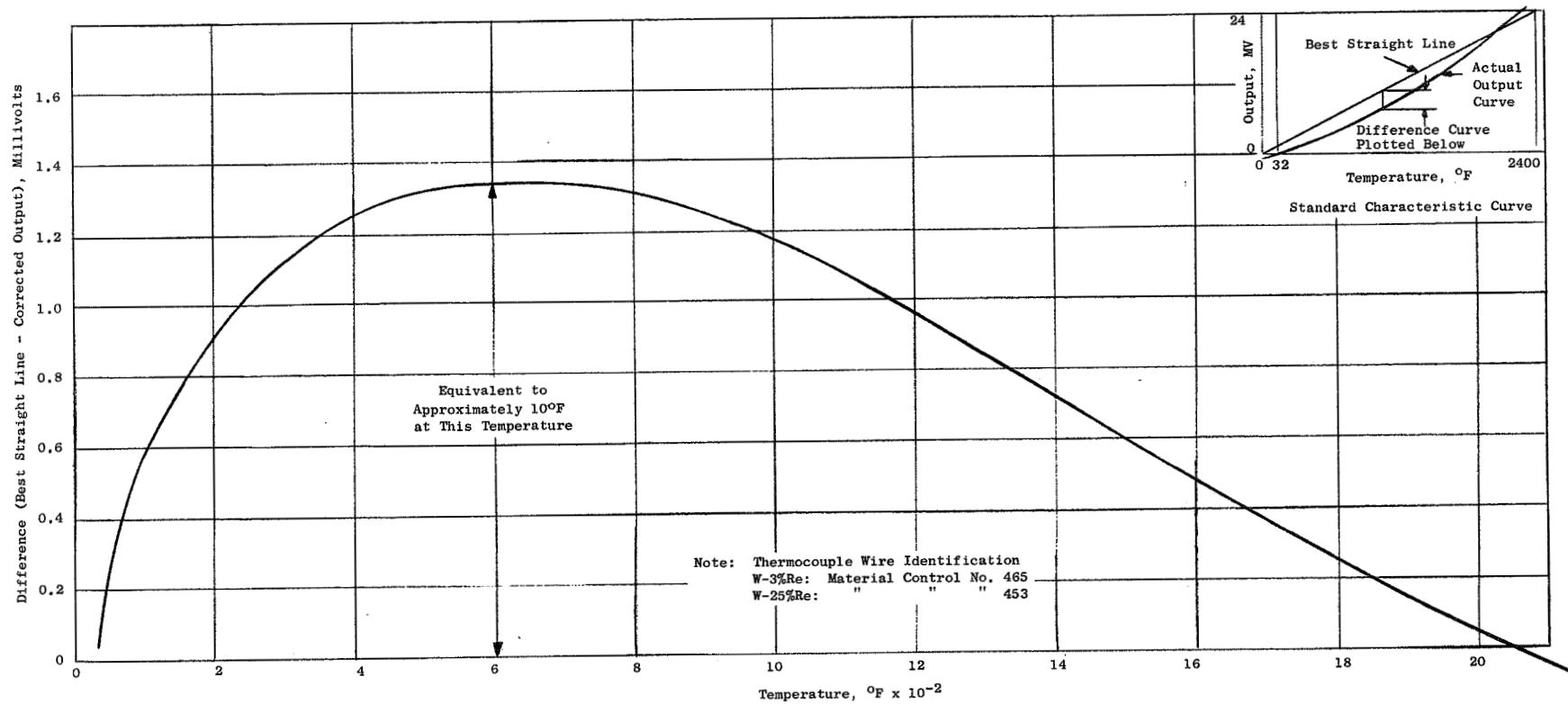


Figure 24. Difference Curve Versus Temperature Obtained in Calibration of Two W-3Re/W-25Re Thermocouples Made From Wire Used to Instrument the T-111 Rankine System Corrosion Test Loop.

TABLE XI

## CALIBRATION DATA FOR W-3Re/W-25Re THERMOCOUPLE WIRE

Temperature °F	Millivolts Below Best <sup>(1)</sup> Straight Line	Millivolts Above Best <sup>(1)</sup> Straight Line	Total Output in Millivolts with 32°F Reference Junction
32.0	0.320		0
100.0	0.587		0.413
160.0	0.790		0.810
200	0.908		1.092
250	1.028		1.472
300	1.126		1.874
350	1.196		2.304
400	1.253		2.747
450	1.298		3.202
480	1.315		3.485
510	1.324		3.776
610	1.338		4.762
670	1.339		5.361
730	1.330		5.970
790	1.313		6.587
830	1.296		7.004
900	1.251		7.749
950	1.215		8.285
1000	1.173		8.827
1100	1.073		9.927
1200	0.961		11.039
1300	0.840		12.160
1450	0.662		13.838
1650	0.416		16.084
1750	0.300		17.200
1850	0.193		18.307
1950	0.092		19.408
2050	0		20.500
2150	--	0.086	21.586
2250	--	0.167	22.667
2350	--	0.240	23.740

(1) Defined by coordinates (millivolts, temperature), (0,0) (24,2400).

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